Study on the effect of the effect of different raw materials sources on spinelization and densification of MgO-Al_2O_3 spinel

A thesis submitted in partial fulfillment of the requirements for the degree of

Bachelor of Technology

In

Ceramic Engineering

By

Sachin Sahoo

ROLL NO: **110CR0106**

Under the Guidance of

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Ceramic Engineering

National Institute of Technology, Rourkela

2014

National Institute of Technology
Rourkela, Odisha, India

Certificate

This is to certify that the thesis entitled "S**tudy on the effect of different raw material sources on spinelisation and densification of magnesium aluminate spinel**" submitted by Mr Sachin Sahoo (110CR0106) in partial requirements of the award of Bachelor of Technology degree in Ceramic Engineering at National Institute of Technology Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge the matter embodied in this thesis has not been submitted to any other institute/university for the award of any degree or diploma.

Date: 09/5/14

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Finally, I am grateful to my parents, all the lab assistants, PhD and M.Tech scholars and last but not the least, my friends who encouraged and helped me during the total period of time and without whose support my project would not have been complete.

Sachin Sahoo

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Date:- 9th May 2014

CONTENTS

Abstract

In our work study is done on the effect of spinelization using different commercially available raw materials of magnesia and alumina. In the study 16-SG, and CL-370 grade of alumina used, and fused magnesia, Netherland magnesia and chemical grade magnesia are used. The spinelization is done using solid state reaction of stoichiometry magnesia and alumina ratio. All the possible combustion of magnesia and alumina are used, which formed 6 batch. These batches are palletized and fired at and fired at 5 different temperature of 1200°C, 1300°C, 1400°C, 1500°C, and 1600°C. The samples are taken for XRD analysis and dilatometer test of the sample determine the off set of sintering and formation of spinelization temperature. The bulk density of the sample is determined. For determining microstructure of spinel SEM of fractured surface is done. And all the experimental data has been studied and correlated with each other. In this study we observed A-16Sg and Netherland magnesia gave highest density of 3.05g/cm^3.

Introduction

Magnesium aluminate spinel ($MgAl_2O_4$) is an excellent refractory oxide of immense technological importance as a structural ceramic. It possesses useful physical, chemical and thermal properties, both at normal and elevated temperatures. It melts congruently at 2135°C, shows high resistance to attack by most of the acids and alkalis and has low electrical losses. Due to these desirable properties, it has a wide range of application in structural, chemical, optical and electrical industry. It is used as a refractory in lining of steel-making furnaces, transition and burning zones of cement rotary kilns, checker work of the glass furnace regenerators, sidewalls and bottom of the steel ladles, glass furnaces and melting tanks.

Typical properties of magnesia alumina spinel

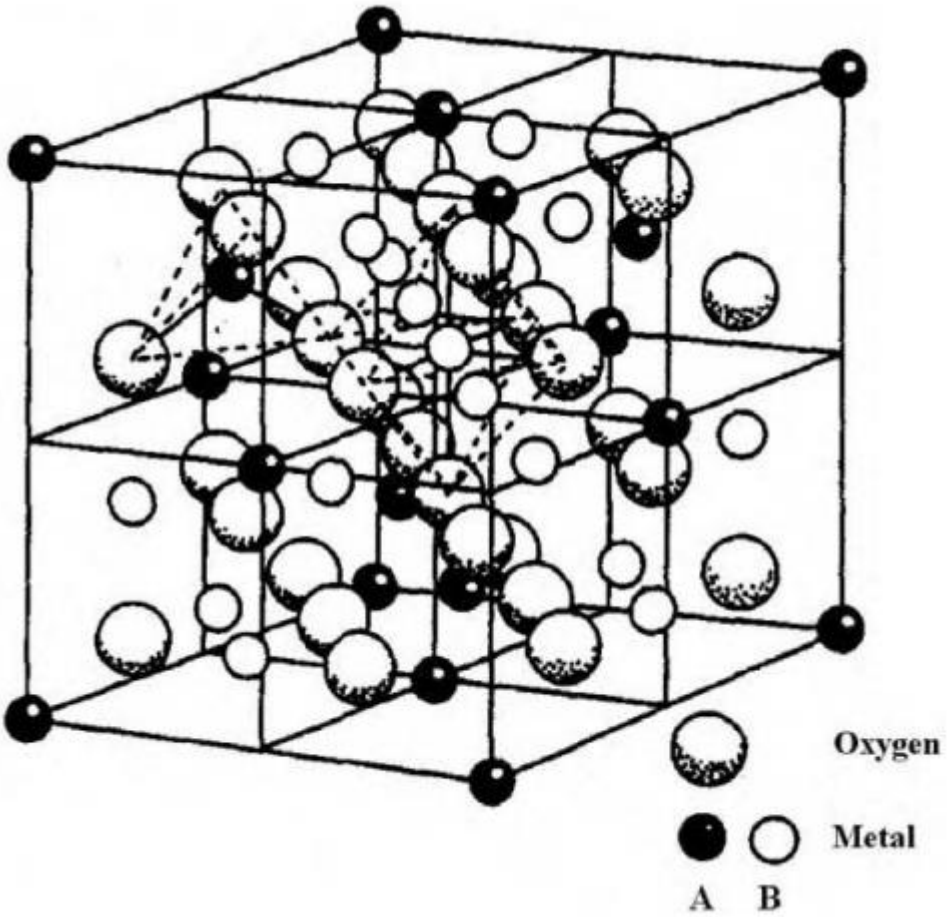
Spinel has a higher melting temperature (2135℃) than Al_2O_3 (2054℃) but lower than MgO (2850℃). Its thermal expansion coefficient ($8.4x10^{-6}$/K) is close to that of alumina ($8.8x10^{-6}$/K), but much lower than MgO ($13.5x10^{-6}$/K). Furthermore, it has superior hydration resistance than periclase and thus can be used in water-based castable systems.

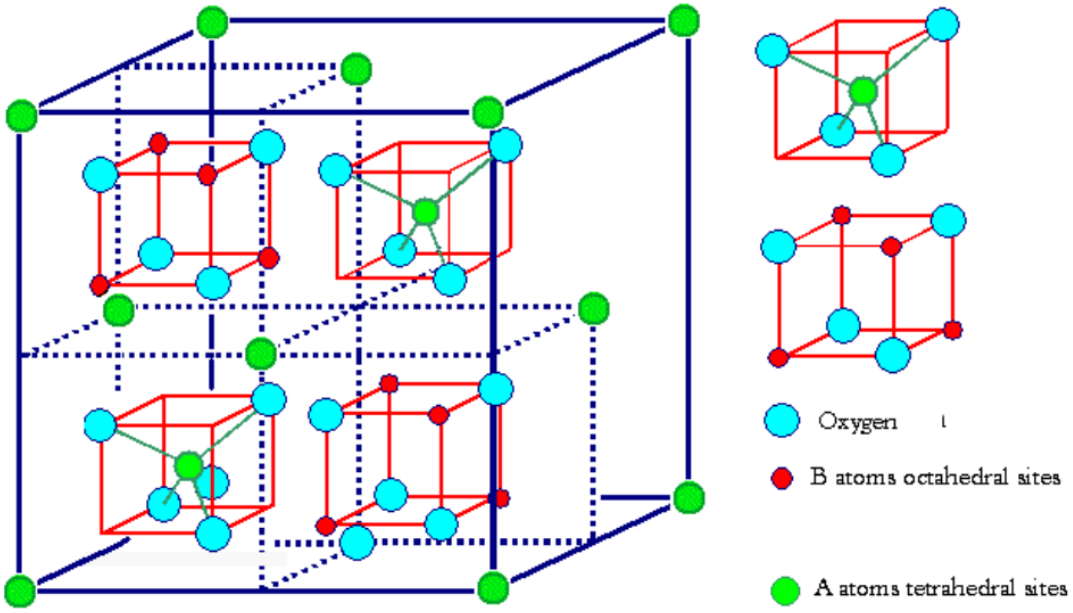
MA Spinel	
Melting point (°C)	2135
Thermal expansion ($\times 10^{-6}$/°C)	
100°C	5.6
500°C	7.6
1000°C	8.4
1500°C	10.2
Thermal conductivity (W/mK)	
25°C	15
100°C	13
500°C	8
1000°C	5
Density ($g \cdot cm^{-3}$)	3.58
Young's modulus (GPa)	240–284
Bending strength (MPa),	
RT	110–245
1400°C	8–10
Hardness (GPa)	15

Spinel crystallography

The crystallographic structure of the magnesia alumina spinel was determined independently by Bragg (1) and Nishikawa (2). The oxygen ions form an almost perfect cubic close-packed array with the metal ions distributed among the resulting positions of fourfold and six fold oxygen coordination. The maximum number of possible tetrahedral and octahedral sites is 64 and 32, respectively.

The 32 oxygen ions occupy the 32-fold positions, and the cations occupy the 8-fold and 16-fold positions. In $MgAl_2O_4$, 8 Mg^{+2} ions are distributed at the 8 positions, and the 16 Al^{+3} ions at the 16 positions, so only 1/8 of the tetrahedral cation sites are occupied by Mg^{+2} ions and 1/2 of the tetrahedral cation sites by Al^{+3}ions. Thus, the MA spinel structure has great potential to accommodate a number of other types of divalent and trivalent cations, allowing large deviations from stoichiometry and solid solution formation. The divalent and trivalent cations are usually limited to those with radii from 0.044 –0.100 nm so that they can be accommodated in the tetrahedral and octahedral sites. (3)

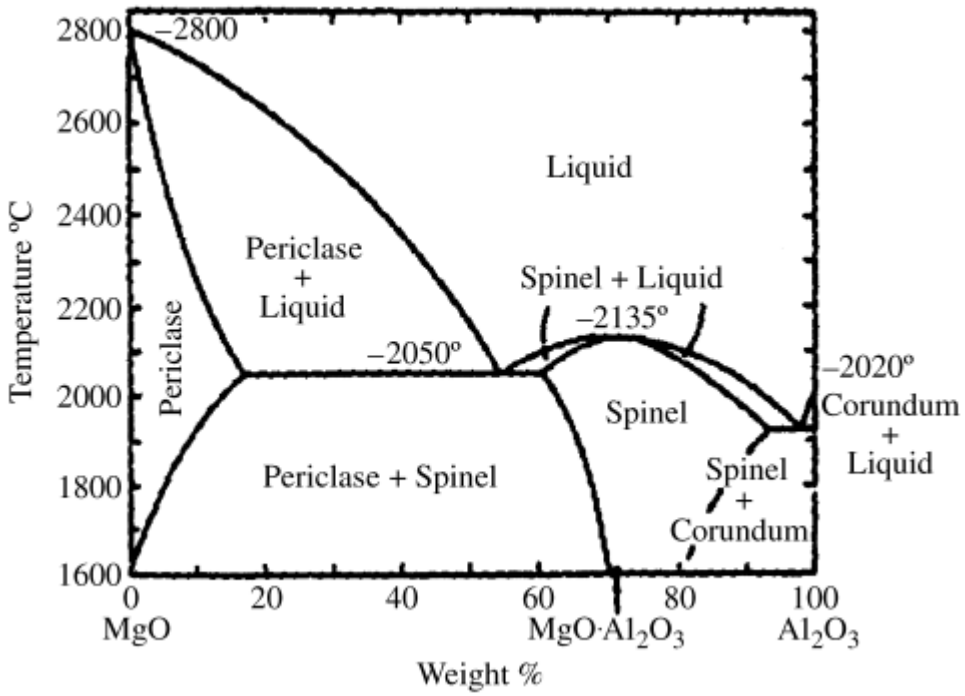




Normal Spinel structure

Magnesia alumina spinel stoichiometry

Magnesia alumina spinel has a range of stoichiometry discernable by its phase field in the MgO– Al_2O_3 phase diagram. Stoichiometric magnesia alumina spinel contains 28.3 wt. % MgO and 71.7 wt. % Al_2O_3. However, with increasing temperature, a wide range of non-stoichiometry may form in the system and the solid solubility of alumina in magnesia alumina spinel is higher than that of magnesia at the same temperature. For example, the solid solubility of MgO and Al_2O_3 at 1600℃ are 2 and 6 wt. %, respectively, but increase to 3 and 10 wt. %, respectively, at 1700℃. Clearly, it is more likely that single-phase magnesia alumina spinel is alumina-rich, and this can be indicated by the notation MgO. nAl2O3wheren, the number of moles of alumina, can be as high as 7.3 (4). MgO-rich spinel (n<1) is, according to the phase diagram, theoretically achievable by quenching from very high temperatures (>1600℃), but more commonly MgO-rich spinel grain contains periclase and is located in the MgO-spinel binary phase field.



Preparation of spinel powders and aggregates

Spinel can be introduced into refractories in the following ways:

- As preformed synthetic spinel powders in the matrix;
- As preformed spinel aggregates;
- By formation of in situ spinel by addition of $Al_2O_3$to MgO-based refractories or MgO to Al_2O_3-based refractories; and
- By formation of in situ spinel by addition of a mixture of MgO and Al_2O_3powders.

Processes for the synthesis of spinel powders or aggregates has been described.

Synthesis of Spinel Powders

- Conventional Oxide Mixing (Solid–Solid Reaction)

MA spinel does not occur in nature and so commercial powders have to be synthesized using a wide variety of techniques. The oldest, simplest, and still most widely used method is the conventional oxide mixing (CMO), or solid – solid reaction, technique. With this technique, powdered MgO- and Al_2O_3-bearing compounds (e.g., oxides, hydroxides, or carbonates) are mixed and pressed into pellets or some other shapes and then heated in a furnace at high temperature for prolonged periods. After synthesis, the product mass is crushed to aggregates or further ground to powders with the desired size distribution. Spinel formation via CMO has been investigated extensively (5) initially, all the elements necessary to form the MA product are present at every interface between particles of MgO and Al_2O_3. Therefore, small crystals with the spinel stoichiometry and structure are nucleated relatively easily on the surfaces of either MgO or Al_2O_3 grains. Once these initial spinel layers form, subsequent growth or thickening of the spinel product becomes much more difficult because, effectively, the two reactants, MgO and Al_2O_3, are no longer in contact but are separated by a rather impenetrable spinel layer. To continue the reaction, a complex counter diffusion process is thus required in which Mg^{+2} ions diffuse away from, and Al^{+3} ions diffuse toward, the MgO– $MgAl_2O_4$ interface and vice versa for the $MgAl_2O_4$–Al_2O_3interface.

To preserve local electro-neutrality during the reaction and throughout the product, it is necessary that, for every three Mg^{+2} ions that diffuse to the right-hand interface, two Al^{+3} ions must diffuse to the left-hand interface. The reactions that occur at the two interfaces may be written, ideally, as

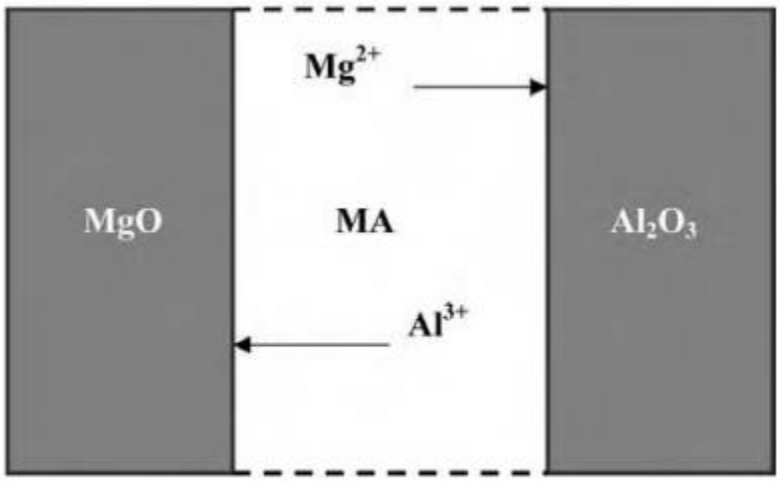
At the $MgO/MgAl_2O_4$ interface:

$$2Al^{+2}\text{-}3Mg^{+2} + 4MgO = MgAl_2O_4$$

At the MgAl2O4=Al2O3 interface:

$$3Mg^{+2}\text{ -}2Al^{+3} + 4Al2O3 = 3MgAl_2O_4$$

The overall reaction is: $4MgO + 4Al2O3 = 4MgAl_2O_4$



Formation of MA spinel product on the reactant MgO and Al_2O_3 grains by a counter-diffusion process.

From which it can be understood that the right-hand interface should move three times as quickly as the left-hand interface.

Spinel formation reaction by this process is particularly slow because ions such as Mg^{+2} and Al^{+3} diffuse slowly. Defects are required, particularly vacant sites into which adjacent ions can hop. High temperatures are also required so that ions have sufficient thermal

energy to, occasionally, vibrate or hop out of one site into an adjacent vacancy or interstitial site. Consequently, it can be difficult for solid-state spinel formation reactions to proceed to completion as the remaining reactants become increasingly separated from each other. A popular way of accelerating the reactions is to frequently regrind the partially reacted mixtures, which acts to break up reactant– product interfaces and to bring fresh reactant surfaces into contact. Alternatively, if gas- or liquid-phase assisted transport of matter can occur, the reactants may be brought together without the need for long-range solid-state diffusion. A small amount of gaseous or liquid transporting agent (mineralizer) may be effective in enhancing reaction rates. Effective mineralizers for spinel formation reactions include fluorine- and boron-containing compounds. The former includes LiF, NaF, AlF_3, Na_3AlF_6, Formation of magnesia alumina spinel product on the reactant MgO and Al_2O_3 grains by acounter-diffusion process. ZnF_2, CaF_2, BaF_2, $CaB_4O_7$and B_2O_3. These mineralizers can form liquid phases and/or help create cation vacancies, increasing spinel formation rate and decreasing its formation temperature.

Literature review

Most important fabrication process for magnesium aluminate spinel in the solid-state reaction between magnesia and alumina .solid state processing form oxide reactants is extensively used in the preparation of convectional ceramic products because of its simplicity and dependence on the solid state reaction. To make magnesia aluminate spinel more economical, many works studied the prospect of natural raw material for the spinel production .but the presence of impurities in the natural raw material were found to be deleterious on the sintering properties. (6)

Bakker and Lindsay produced a reactive spinel powder from hydroxide material with 15% aluminum fluoride as mineralizer. Reactive spinel was reported to have good properties after sintering and excess aluminum showed deleterious effect.(7)

Using magnesite powder from metallurgical plant and bauxite, Aletseeva and others found little amount of forsterite, corundum, pericalse and montecellite phases in addition to spinel phase on firing between 1100 ℃ to 1600℃ in an oxidizing atmosphere.(8)

Chen and Tian synthesized active magnesium aluminate spinel from light burnt magnesite and alumina by calcination and then studied the sintering behavior, density-porosity relationship and grain growth during sintering. They reported spinel formation at 1000^{o}C by solid phase reaction and found best sintering behavior for the material calcined at 1250^{o}C. (9)

Mazzoni and others did preparation of spinel from active oxides by thermal treatments of $Mg(OH)_2$ and $Al(OH)_3$. Activation was done in impact and friction mill, which reduced the spinel formation temperature. [10]

Xiang and Hug prepared spinel from natural materials with additions of Fe_2O_3, CaO and TiO_2. Expect spinel phase, they also found few silicates, titaneous and ferrous minerals. Mineral composition was found to be changed with the different oxide additions. [11]

Xiang and others also prepared spinel from natural magnesite and bauxite with different proportion of magnesia and alumina. They observed that spinel formation started around 960^{o}C to 1000^{o}C, which completed between 1300^{o}C and 1350^{o}C. The final body consists of 85 to 95 % spinel, 10% silicates and 5% titaneous materials. The authors also reported that this spinel with corundum showed better slag resistance in castable applications. [12]

Degtyareva and others prepared spinel using magnesite and by products of chemical industry as source of alumina. The by-products were consisted of 73-78% alumina and rest chromia and silica. They found increasing amount of magnesite decreased the free Al_2O_3-Cr_2O_3 phase and at 33% of magnesite only and forsterite phases were present higher amount of magnesite resulted in free periclase phase. [13]

Sharipo and others used magnesite and by products of chemical industries as a source of alumina, is used to produce fused spinel. They used 67% of the waste material and 33 % of magnesite and then fused the mixture in an electric arc furnace at 2400^{o}C. They reported reduced iron oxide in the fused body and recommended the fused body mass for the production of spinel brick. [14]

EXPERIMENTAL PROCEDURE

Raw material

For preparation of spinel is done using three different grade of magnesia and two different grade of alumina. They are:-

Alumina

- A-16SG

  This grade of alumina is a product of Almatis premium reactive alumina have specially tailored grain sizes to meet the fired density and shrinkage Requirement for precision ceramics. The fully ground Reactive alumina are specially designed for the production of high performance refractories where defined particle packing, rheology and consistent placement characteristics are as important as superior physical properties of the final product.

  Chemical composition:-

  Al_2O_3→ 99.8% CaO→ 0-0.05%

  SiO_2 → 0-0.05% B_2O_3→ 0-0.006%

  MgO→ 0-0.06% Fe_2O_3→ 0.03%

  Na_2O→ 0.10%

- CL-370

  This is a product of Almatis, it is calcined alumina. Due to the excellent high temperature properties of alpha-alumina, Calcined Aluminas are utilized in many refractory applications, both as monolithic and shaped products. Depending on the particle size distribution and crystal size, Calcined Aluminas serve a variety of functions in refractory.

  Chemical composition:-

  Al_2O_3→ 99.7% CaO→ 0-0.02%

  SiO_2 → 0.03-0.07% B_2O_3→ ------

  MgO→ ------ Fe_2O_3→ 0.03%

  Na_2O→ 0.10-0.14%

Magnesia

- Nedmag 200

  NedMag is a high purity synthetic Dead Burned Magnesia used for the production of basic refractories.

  Chemical composition

  MgO→ 97.00% CaO→ 0.74-0.9%

  SiO_2 → 0.12-0.2% B_2O_3→ 0.01-0.02%

  MnO_2→ 0.12-0.02% Fe_2O_3→ 0.44-0.6%

  Al_2O_3→ 0.7-0.2%

- Fused magnesia (FM 97)

  Crystals fused magnesia uses high purity calcined magnesia as raw material which is fused by electric arc furnace to finish. It has the features such as high purity, dense crystal, good resistance to slag erosion, good thermal shock stability etc.

  Chemical composition:-

  MgO→ 97.00% CaO→ 1.38-1.50%

  SiO_2 → 0.41-0.5% Fe_2O_3→ 0.46-0.7%

  Al_2O_3→ 0.12%

- Chemical grade magnesia

  Chemical grade magnesia which is manufactured by Loba chemicals.

  Chemical composition:-

  MgO→ 98.00% CaO→0.02 %

  Carbonate →1.29-1.5 % Fe_2O_3→0.005 %

Batch preparation

The batch is prepared by mixing 28.3 wt. % MgO and 71.7wt. % Al_2O_3. The batch is prepared by mixing 28.3 wt. % MgO and 71.7wt. % Al_2O_3 in 100ml of iso-propan-2-al. stirring of the batch is done for 60 min in magnetic stirrer, until uniform mixing is complete.

After completion of stirring the mixture is dried in drier until complete removal of iso-propan-2-al. The mixing processes done for all bathes with composition as give.

Batch no	alumina	magnesia
Batch 1	A-16SG	Nedmag200
Batch 2	CL-370	Nedmag200
Batch 3	A-16SG	Fused Magnesia
Batch 4	CL-370	Fused Magnesia
Batch 5	A16 SG	Chemical Grade Magnesia
Batch 6	CL-370	Chemical Grade Magnesia

Sample preparation

For preparation of spinel and testing pallets of 15mm diameter are prepared.

The pallet preparation is done by mixing batch with 4% PVA solution in agate mortar. After uniform mixing the sample is pressed using hydraulic-press. Under pressure of 1000kg/cm^2, the sample is dried. The same process is continued for all 6 batches.

Firing

4 pelleted sample of each batch is fired for at 1200℃, 1300℃, 1400℃, 1500℃ & 1600℃ for 2hrs. Firing is done for each batch sample at five temperature for 2hrs.

Testing

XRD analysis of fired samples:

X-Ray Diffraction was performed using Philips X-Ray diffractometer PW 1730 with nickel filtered Cu K_α radiation (λ=1.5406A^o) set to 4°C per min and scanned continuously in the range of 15° to 60°. The result of XRD data has been analyzed for different phases in the material. The XRD of all the batches and of each batch having different temperature.

Shrinkage calculation

The original sample diameter before sintering was measured as 15 mm. After sintering the pellet diameter was measured and shrinkage was calculated according to the formula

$$\text{Shrinkage} = \frac{(\text{diameter before sintering} - \text{diameter after sintering}) * 100}{\text{diameter before sintering}}$$

Apparent porosity and bulk density calculation

The bulk density and apparent porosity of the sintered pellets were calculated by Archimedes principle using kerosene. Dry Weight is measured and then the pellets were kept in water and then vacuuming is done for about 2 hrs. After that suspended weight is measured using apparatus in which pellet is suspended in water. After the suspended weight, soaked weight is taken. Hence the dry weight, soaked weight and suspended weight were measured accurately. The bulk density and apparent porosity were calculated by using the formulas:

$$\text{Bulk density} = \frac{Dry\ weight}{Soaked\ wt - Suspended\ wt}$$

$$\text{Apparent porosity} = \frac{Soaked\ wt - Dry\ wt}{(Soaked\ wt - Suspended\ wt)\ x\ 100}$$

Scanning electron microscopy

The SEM of fracture surface are done. The SEM will revile the microstructure and details of the material. So SEM of all batches of sintered sample at 1600^{o}C are done.

Result and discussion

The experiment are carried out using different grade of alumina and magnesia.

Alumina

- A-16SG
- CL-370

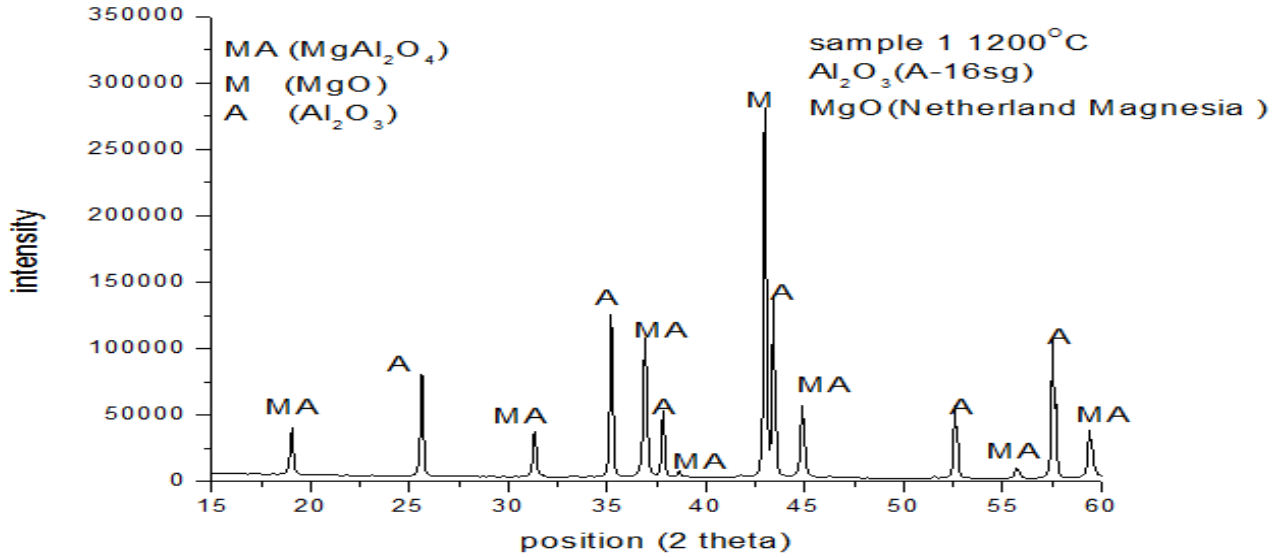
Magnesia

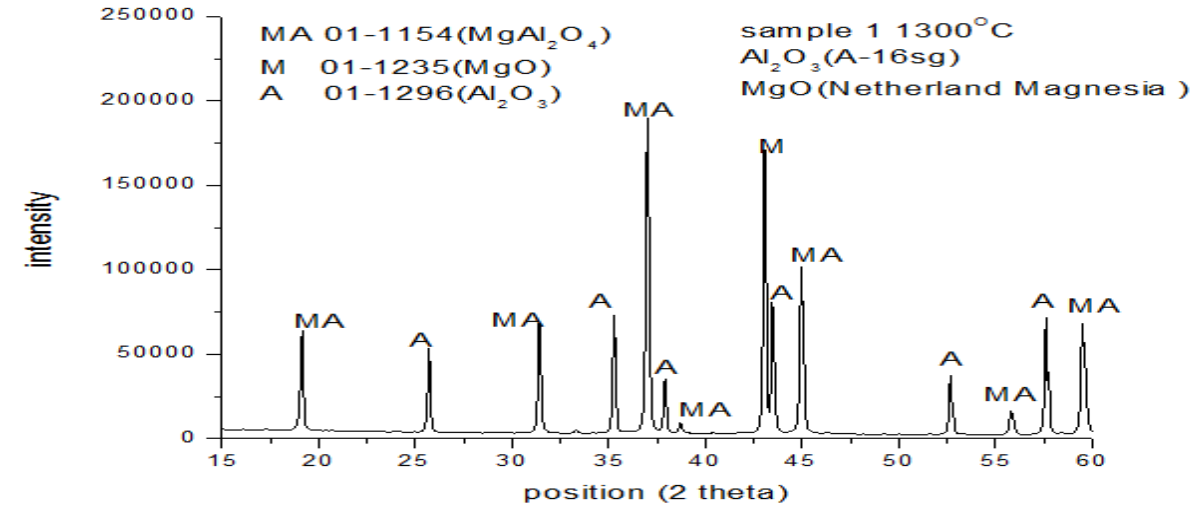
- Nedmag 200
- Fused magnesia (FM 97)
- Chemical grade magnesia

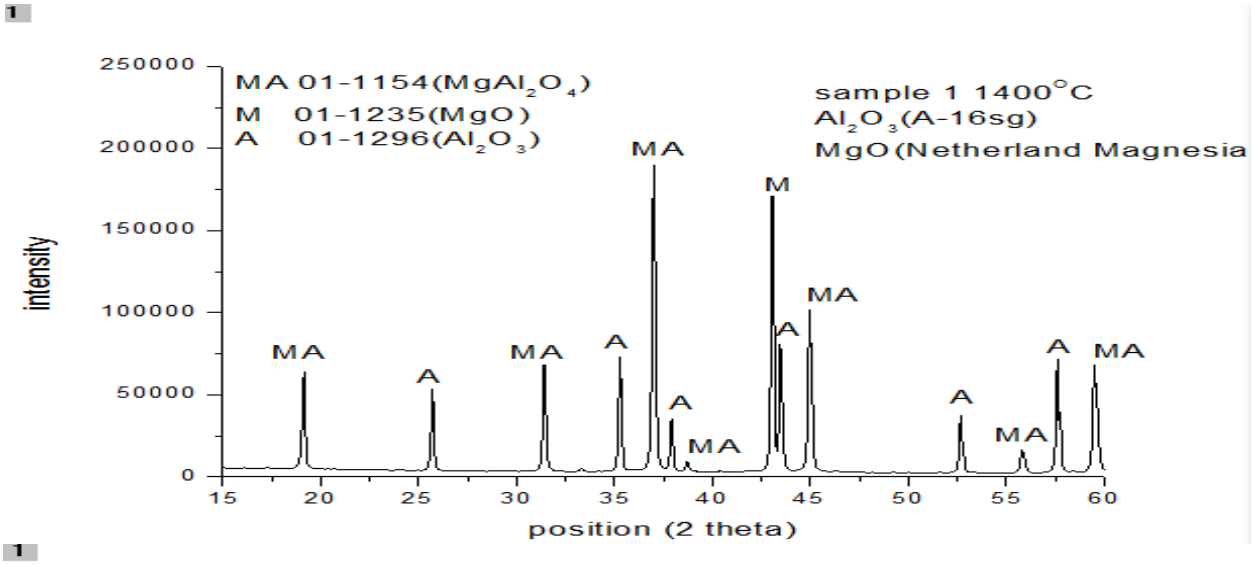
The experiment are carried out step by step using all the six batches and at different firing temperature.

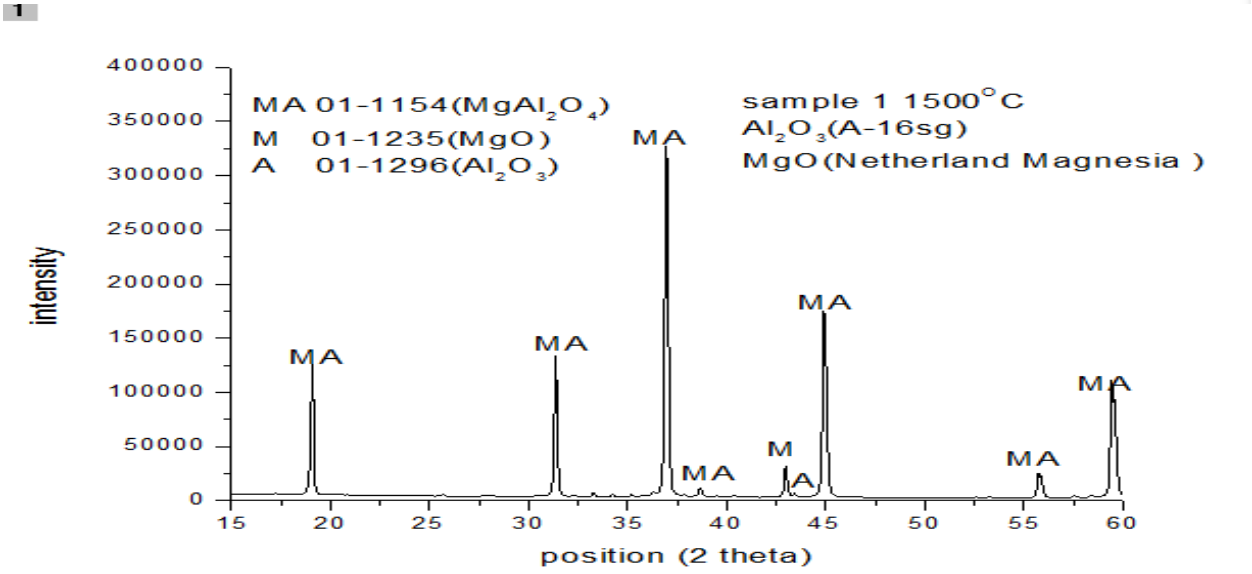
Batch 1 (A-16sg & Nedmag200)

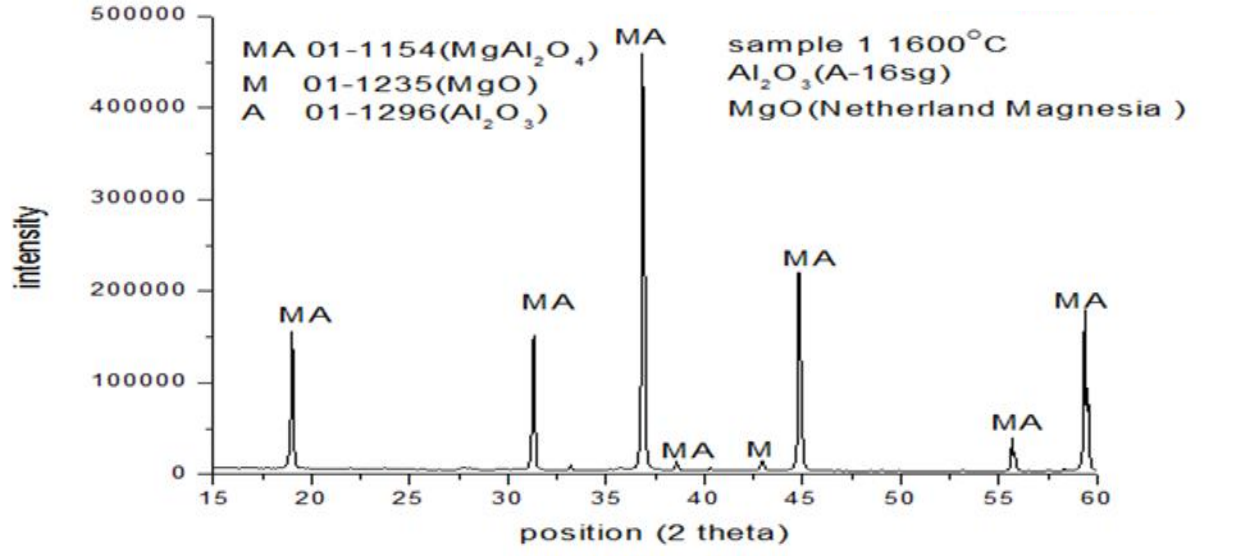
XRD analysis at different temperature





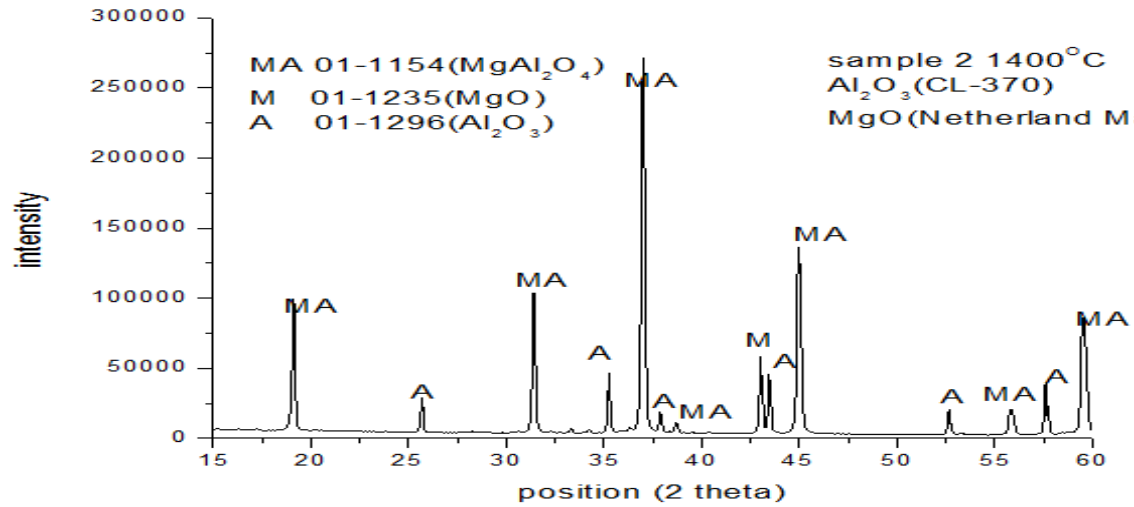

MA 01-1154($MgAl_2O_4$)
M 01-1235(MgO)
A 01-1296(Al_2O_3)
sample 1 1400°C
Al_2O_3(A-16sg)
MgO(Netherland Magnesia
intensity
position (2 theta)

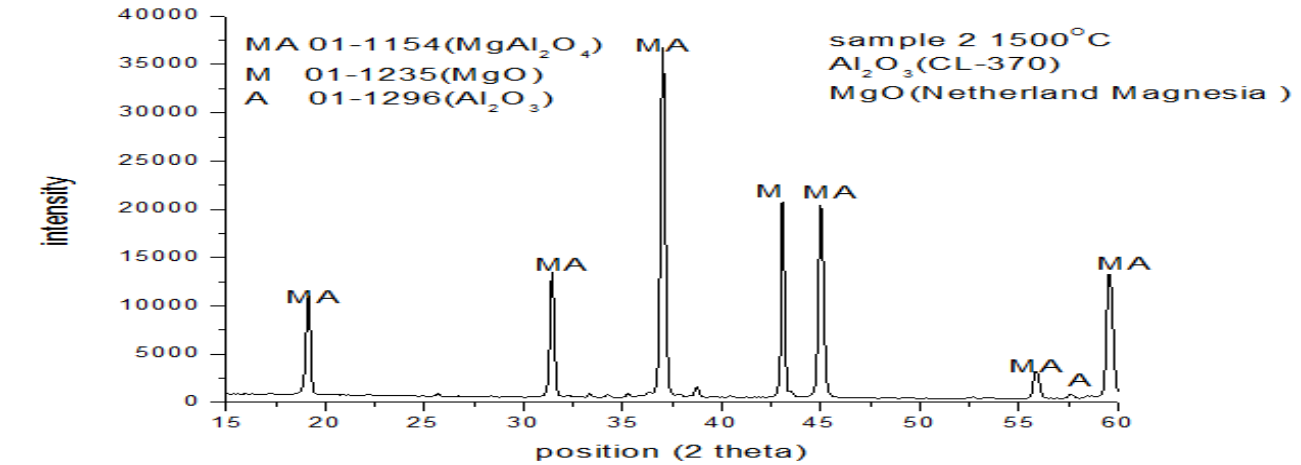

MA 01-1154($MgAl_2O_4$)
M 01-1235(MgO)
A 01-1296(Al_2O_3)
sample 1 1500°C
Al_2O_3(A-16sg)
MgO(Netherland Magnesia)
intensity
position (2 theta)

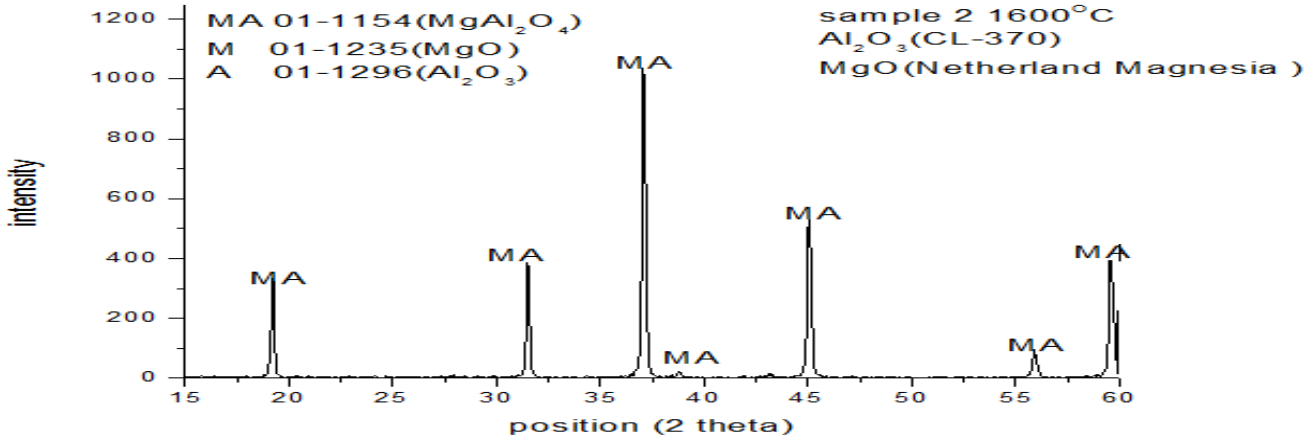

MA 01-1154($MgAl_2O_4$)
M 01-1235(MgO)
A 01-1296(Al_2O_3)
sample 1 1600°C
Al_2O_3(A-16sg)
MgO(Netherland Magnesia)
intensity
position (2 theta)

Batch 2 (CL-370 & Nedmag200)

XRD analysis at different temperature

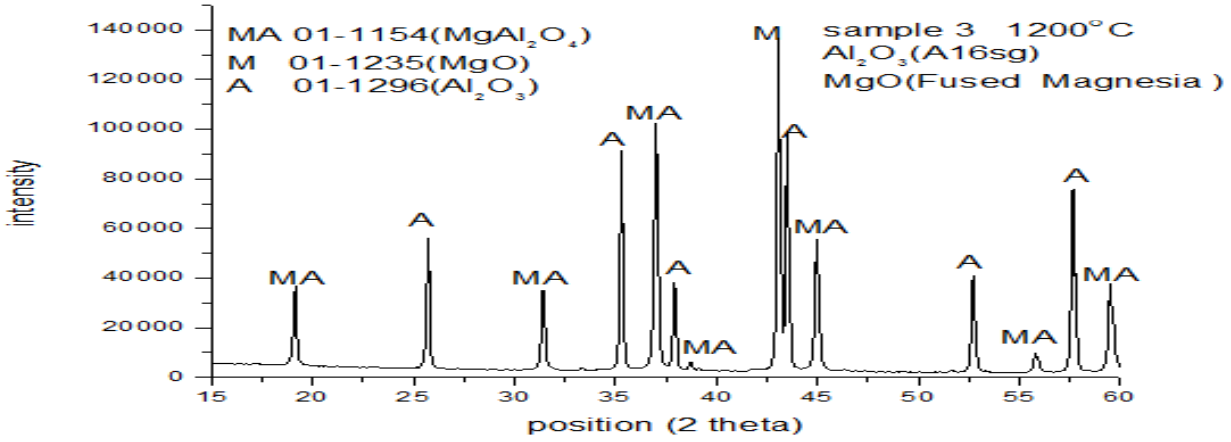


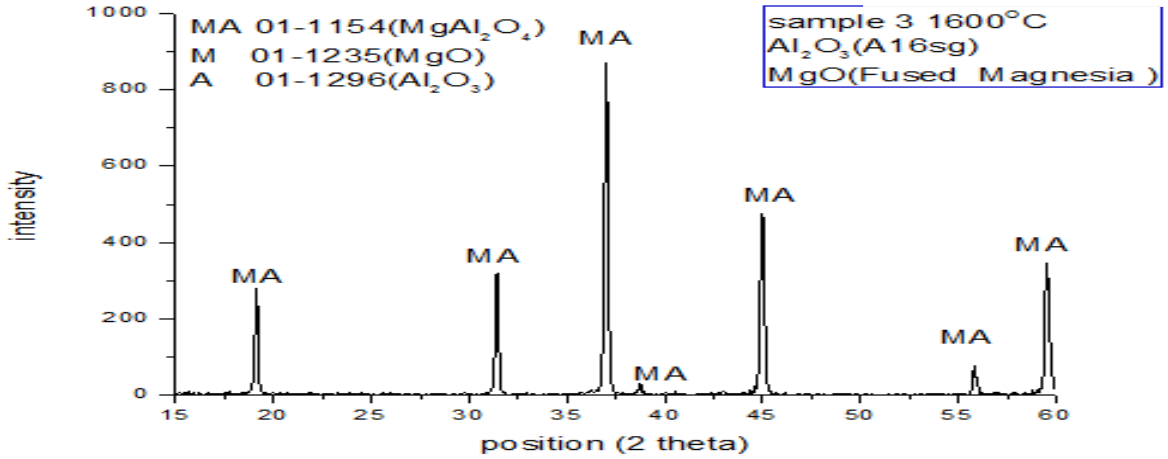


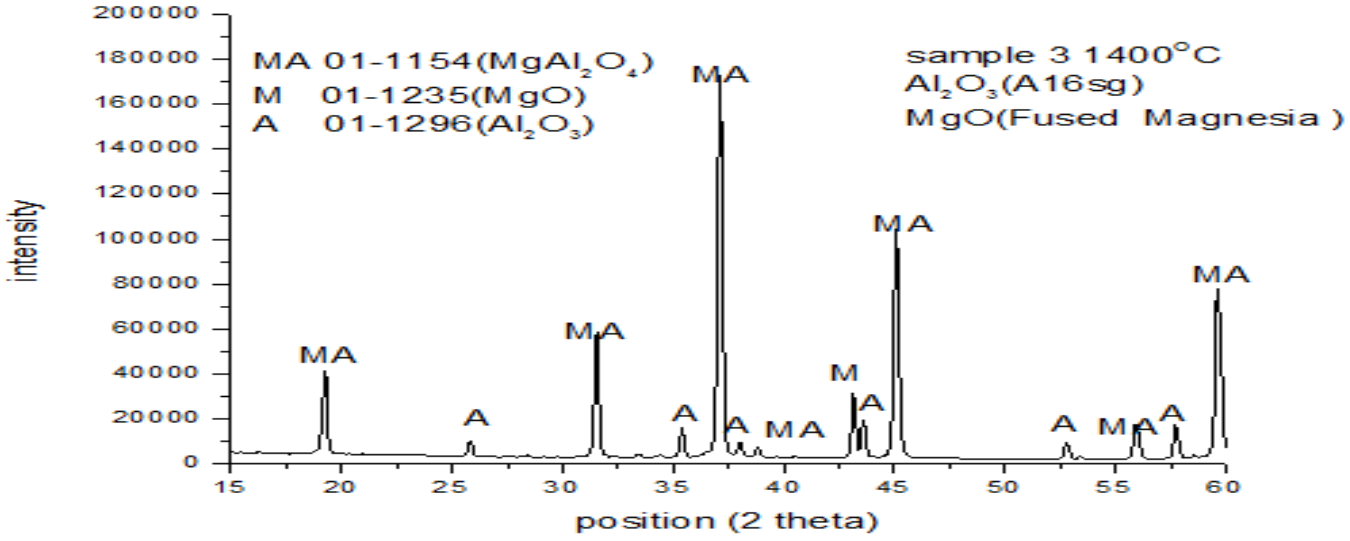


Batch 3(A-16sg & fused magnesia)

XRD analysis at different temperature



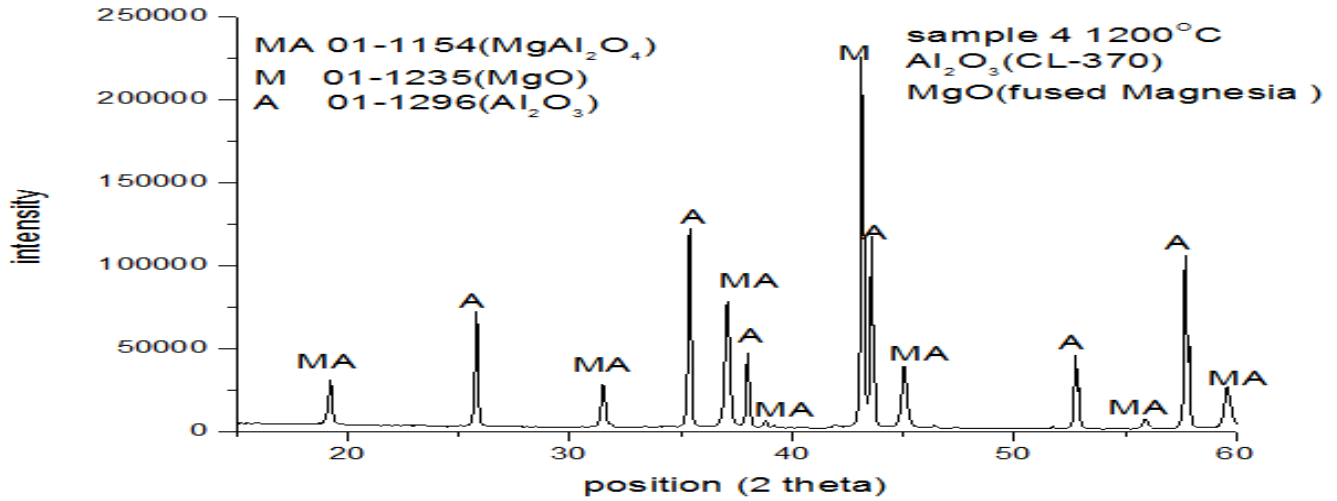


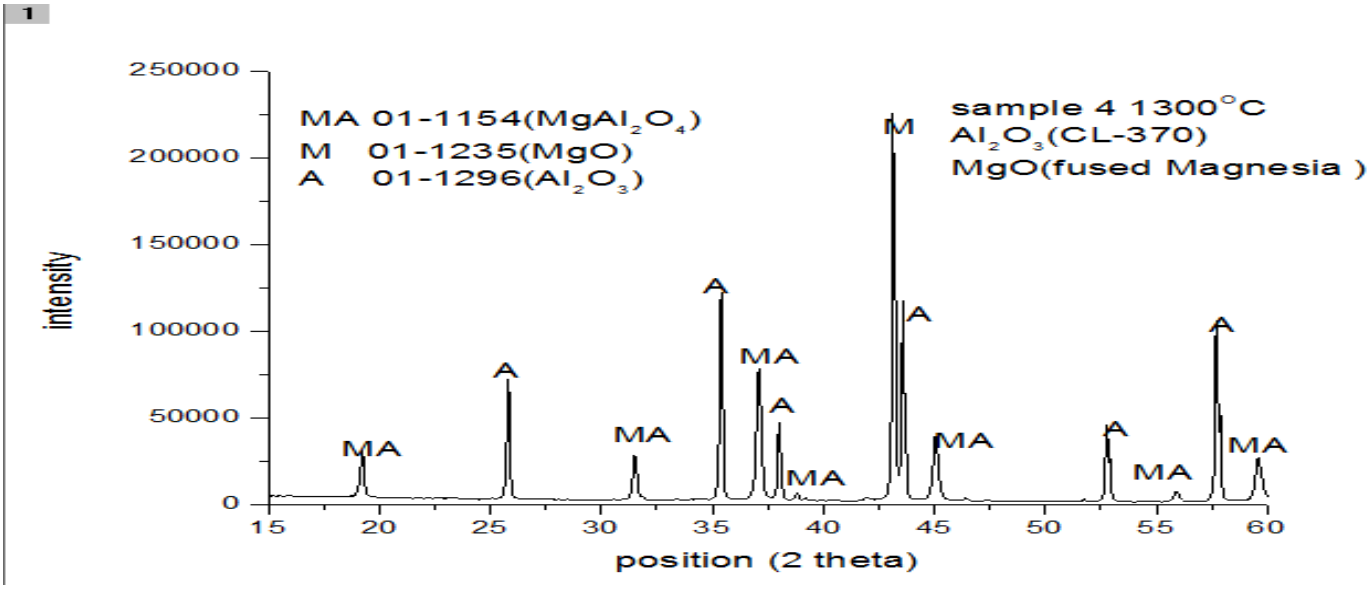


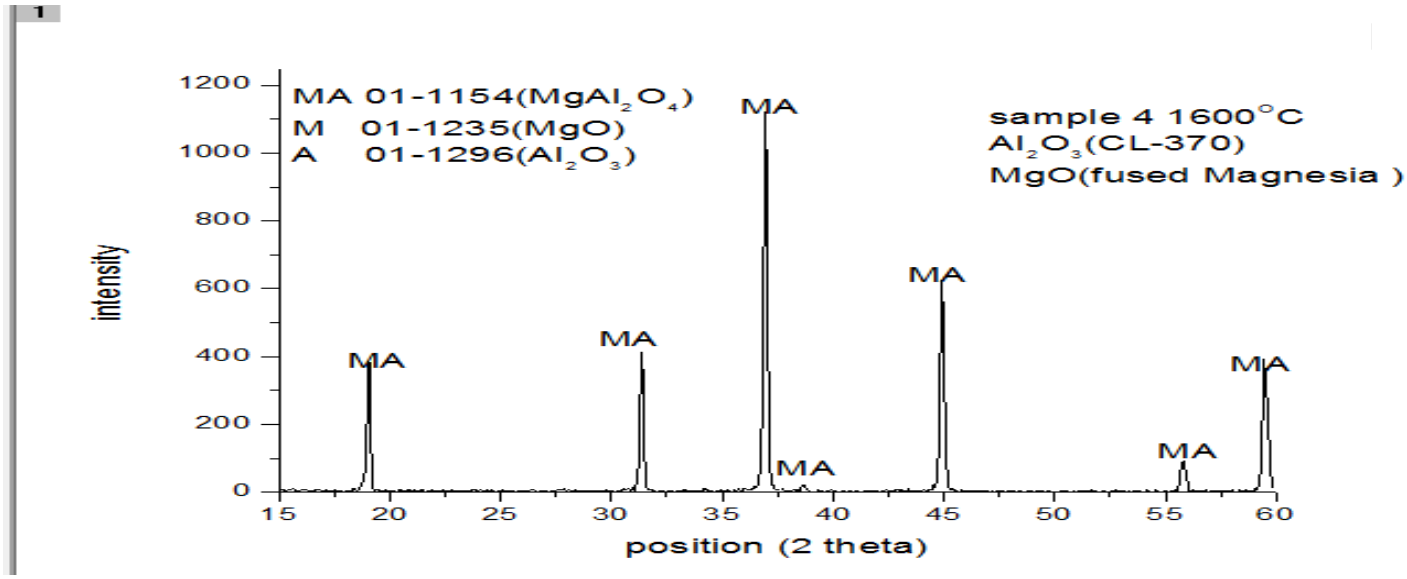
Batch 4(CL-370 & fused magnesia)

XRD analysis at different temperature

]

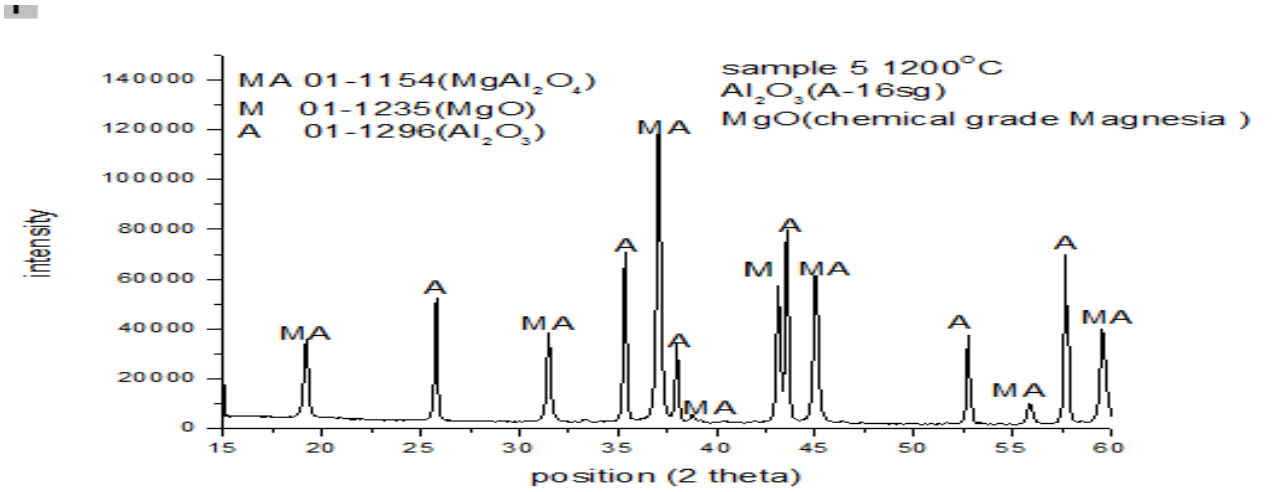


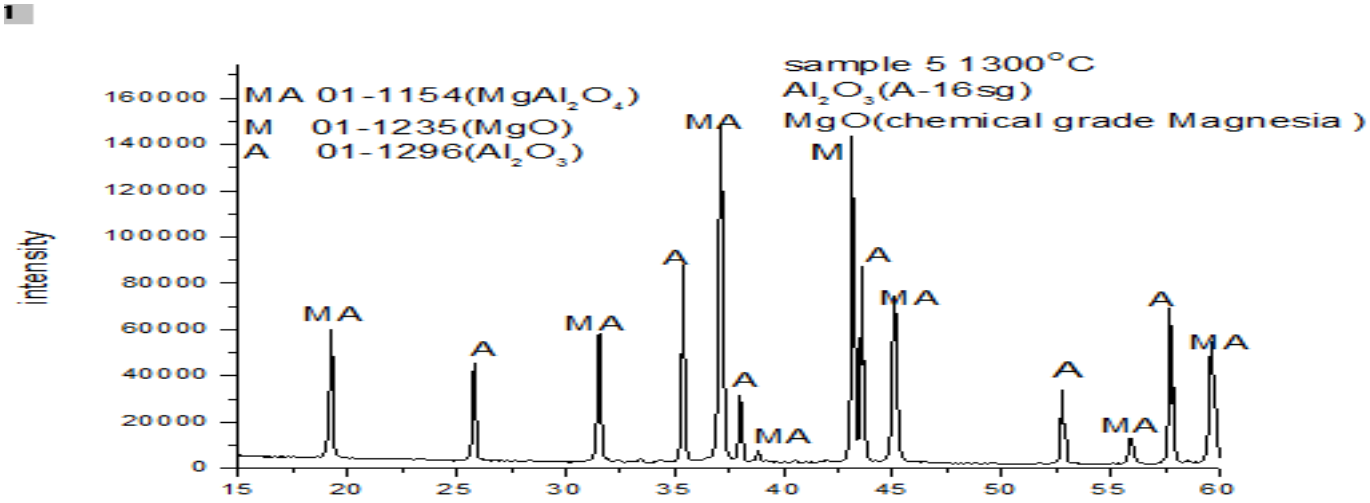


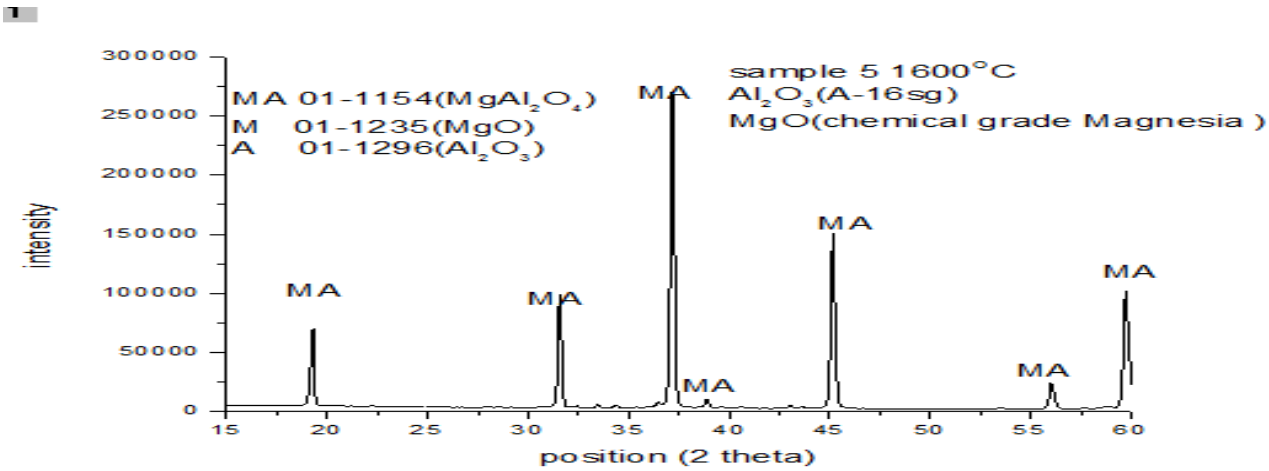


Batch 5 (A16 sg & chemical grade magnesia)

XRD analysis at different temperature

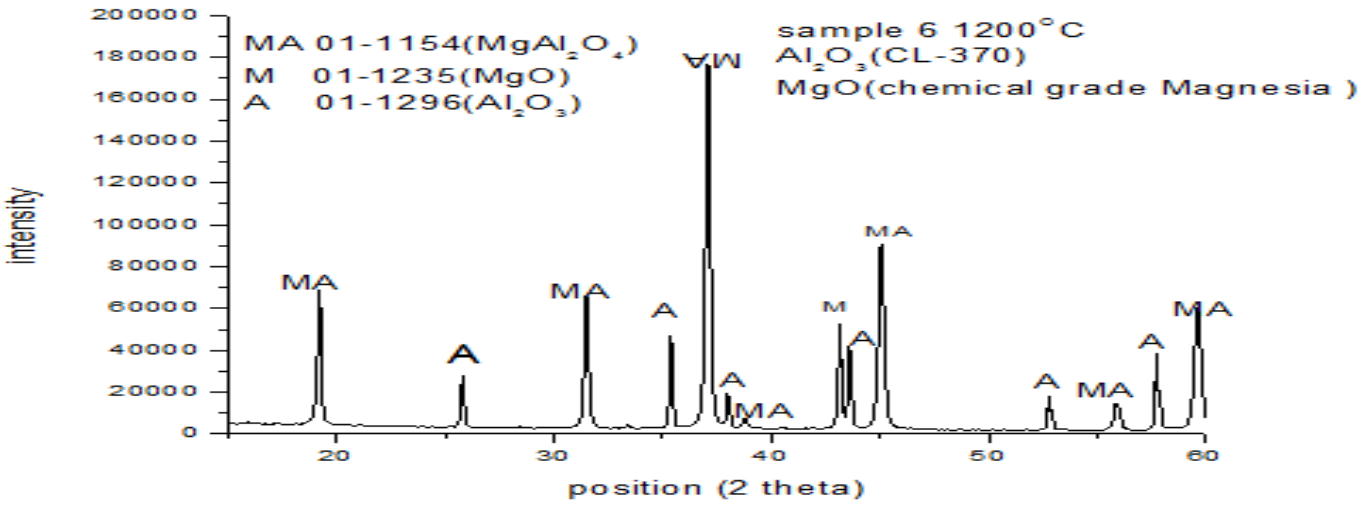


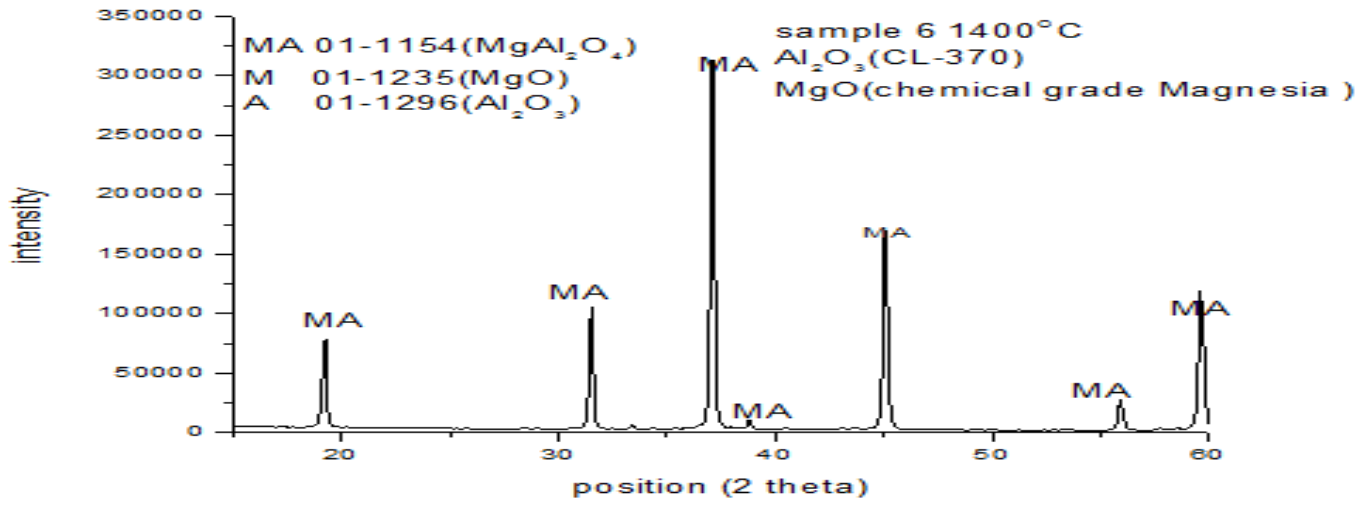


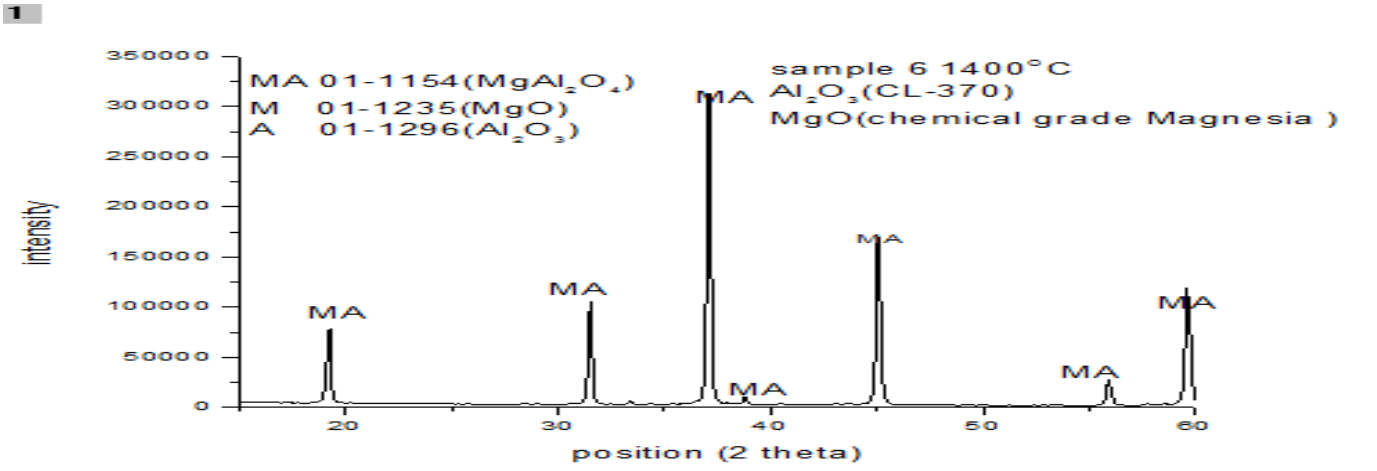


Batch 6 (CL-370 & chemical grade magnesia)

XRD analysis at different temperature





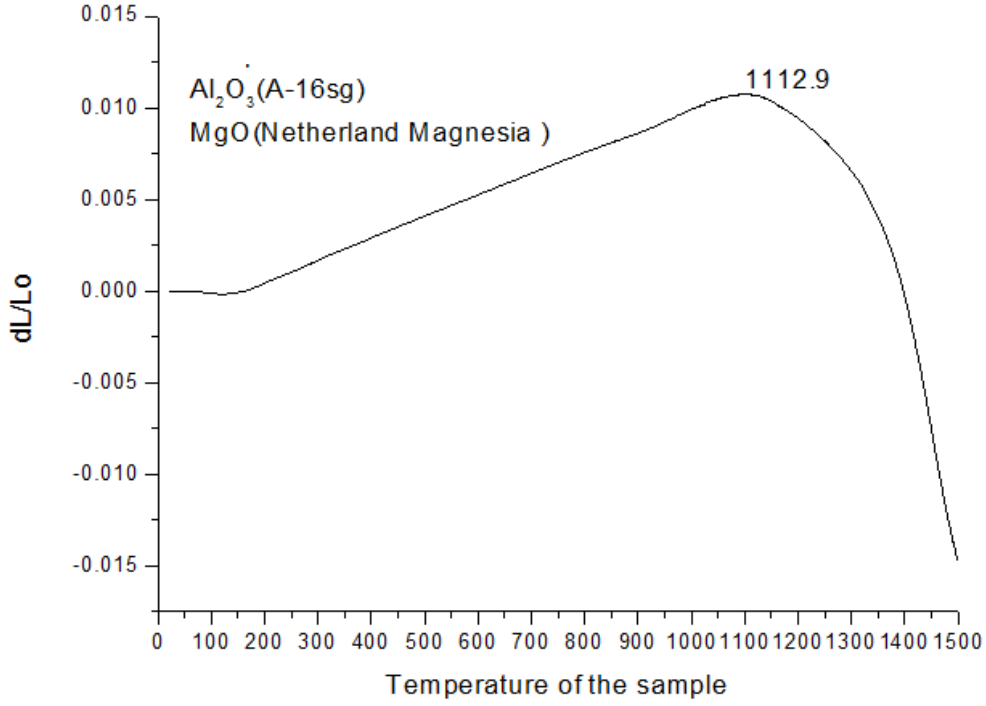


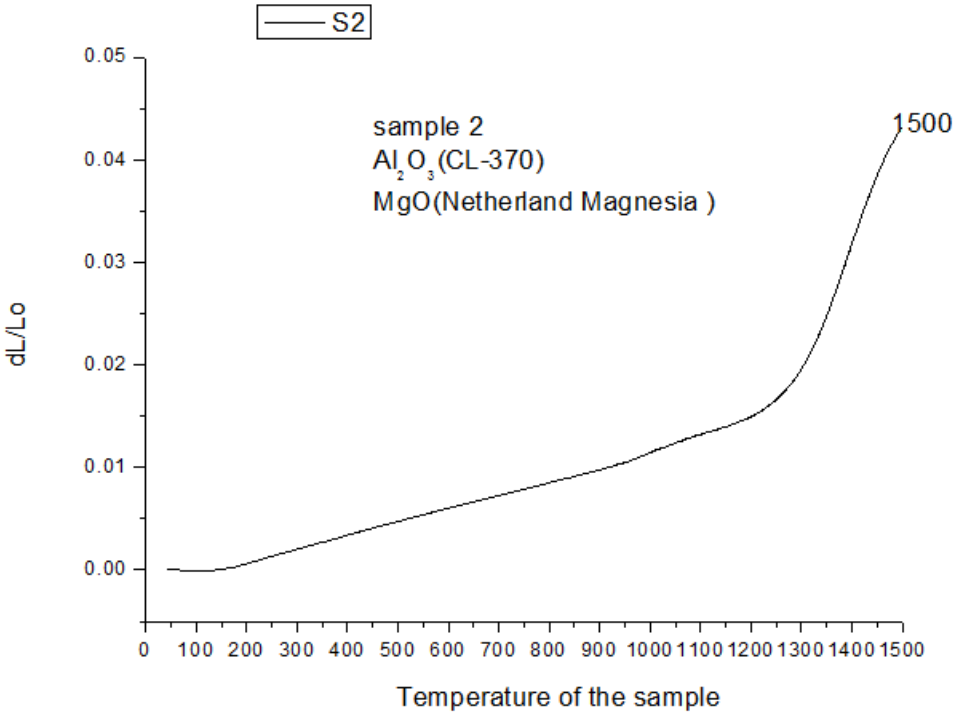
XRD

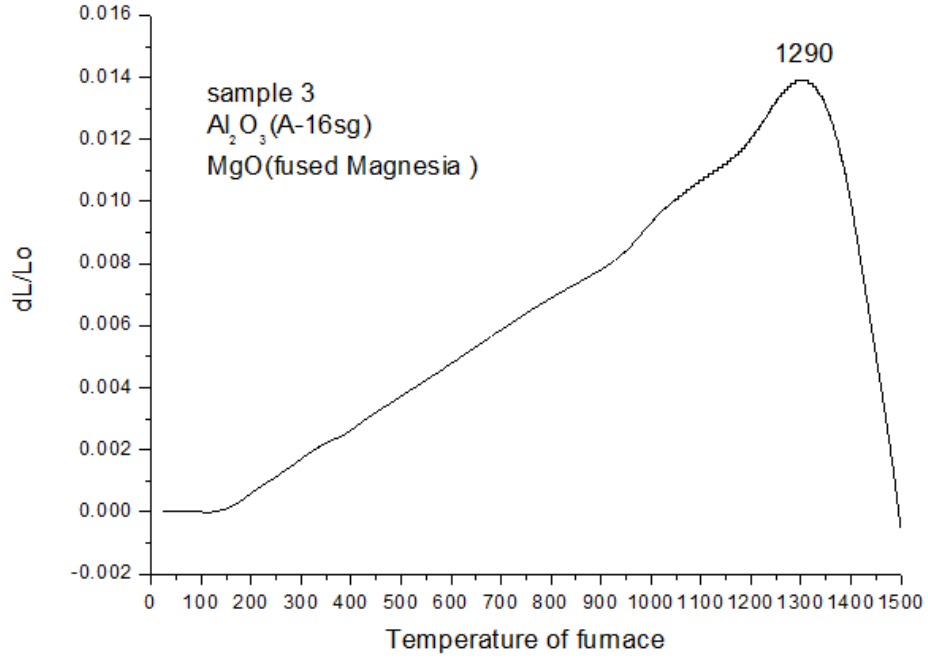
In the above XRD analysis of all the batch, it was found that in all batches. Spinelization has started below 1200℃. Hence spinel peaks were found at 1200℃ -XRD of all the batches of material. The amount of spinel formed increases with increase in temperature. Which can be concluded from increase in intensity of the spinel peak in the XRD plot of all batches with increase in temperature. Complete spinelization is observed in all magnesia alumina batches fired above 1500°C. As there is hardly any magnesia and alumina peak in sample fired above 1500°C. But in batch 5 (A16sg & chemical grade magnesia) & batch 6 (CL-370 & chemical grade magnesia) shows complete spinel formation at 1400°C. This is due to the high surface area and fine particle size of chemical grade magnesia. This reacts with the alumina at relatively lower temperature to form complete spinelizataion as compared to other batches.

Dilatometer testing of batch

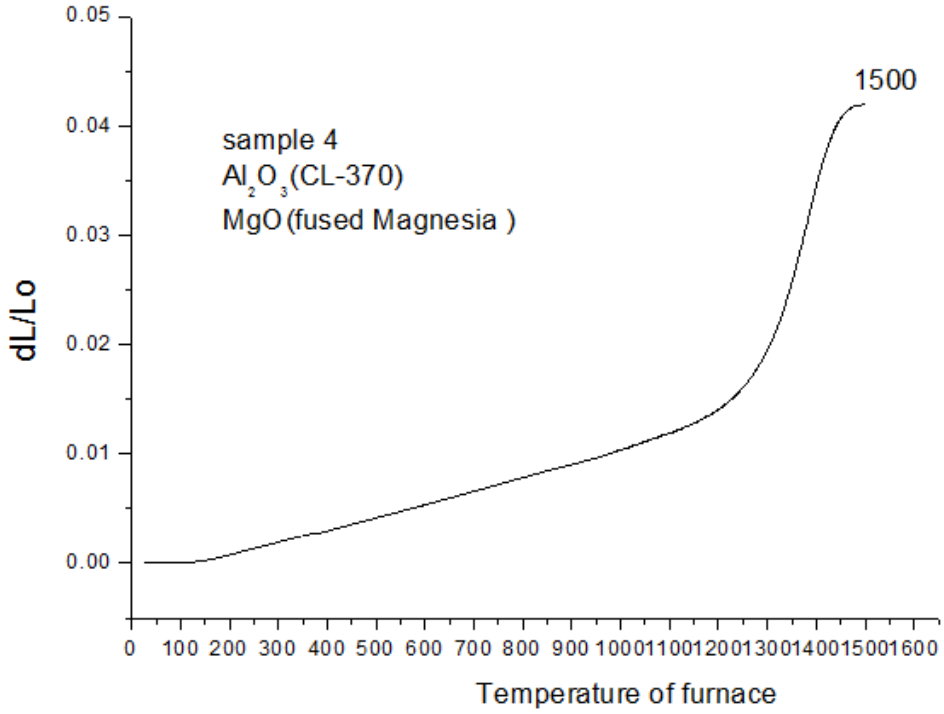
1

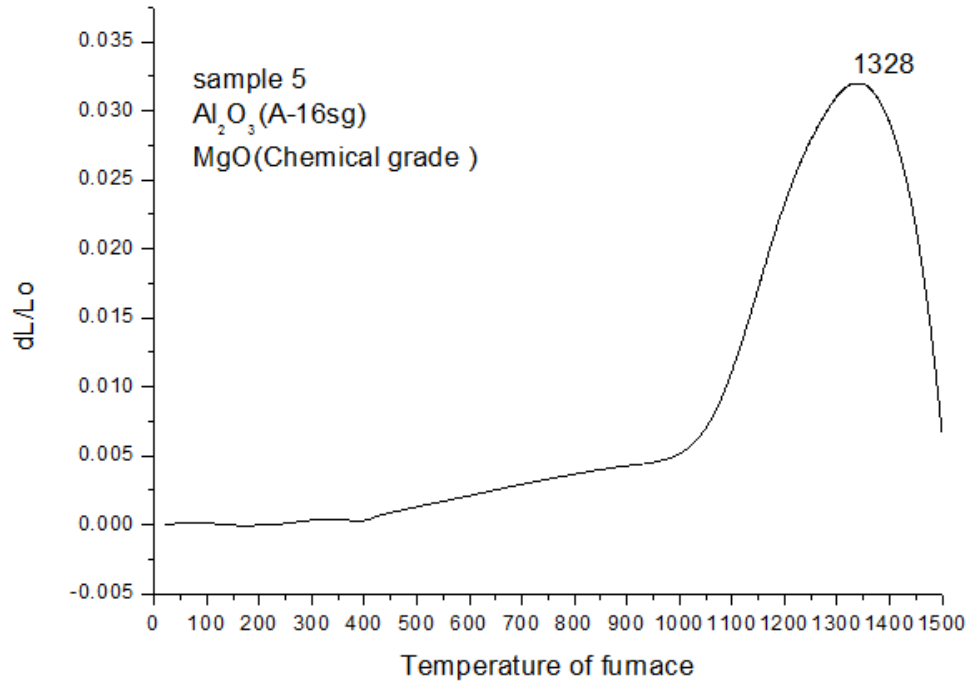


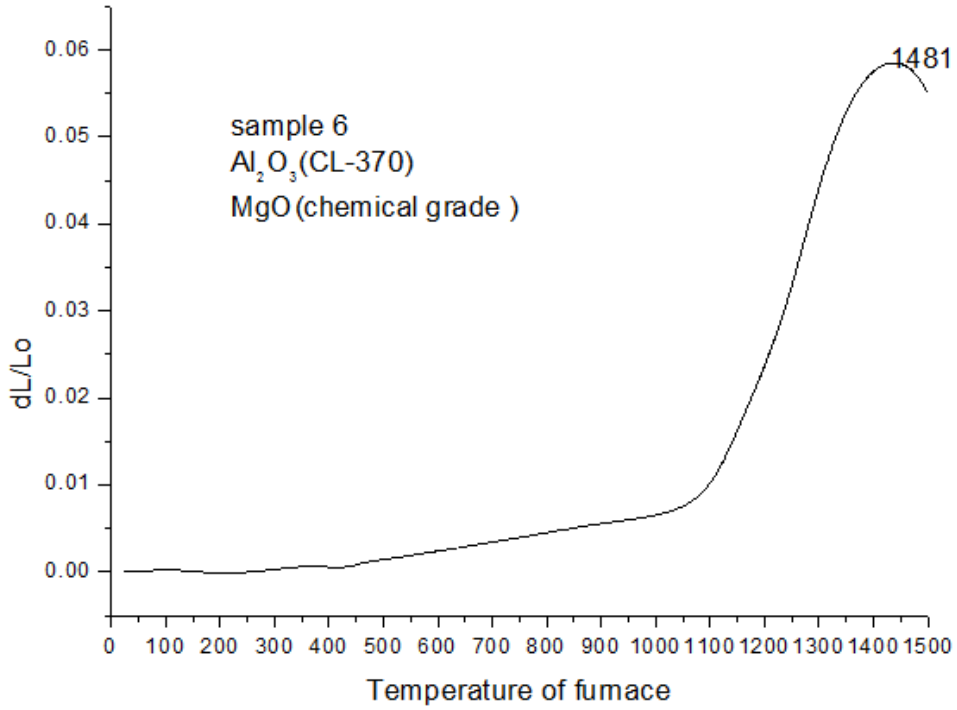



1290
sample 3
Al2O3(A-16sg)
MgO(fused Magnesia)
dL/Lo
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
-0.002
0 100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500
Temperature of furnace

1


1500
sample 4
Al2O3(CL-370)
MgO(fused Magnesia)
dL/Lo
0.05
0.04
0.03
0.02
0.01
0.00
0 100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600
Temperature of furnace


sample 5
Al_2O_3(A-16sg)
MgO(Chemical grade)
1328
dL/Lo
Temperature of furnace


sample 6
Al_2O_3(CL-370)
MgO(chemical grade)
1481
dL/Lo
Temperature of furnace

Dilatometer test

In the dilatometer test we observe that there is initially increase in length with increase in temperature as there is formation of spinel which is associated with expansion of sample. Hence there is increase in length. At high temperature there is also sintering of the sample. On further increase in temperature there is decrease in length where sintering overcomes the expansion effect of the spinel formation. This point gives idea about in which material will have better densification at low temperature.

In Batch 1 (A-16sg & Nedmag200) at 1112.9^{o}C is the temperature at which densification over takes the expansion due to spinel formation. And it has the lowest temperature of all other batch hence it showed maximum density.

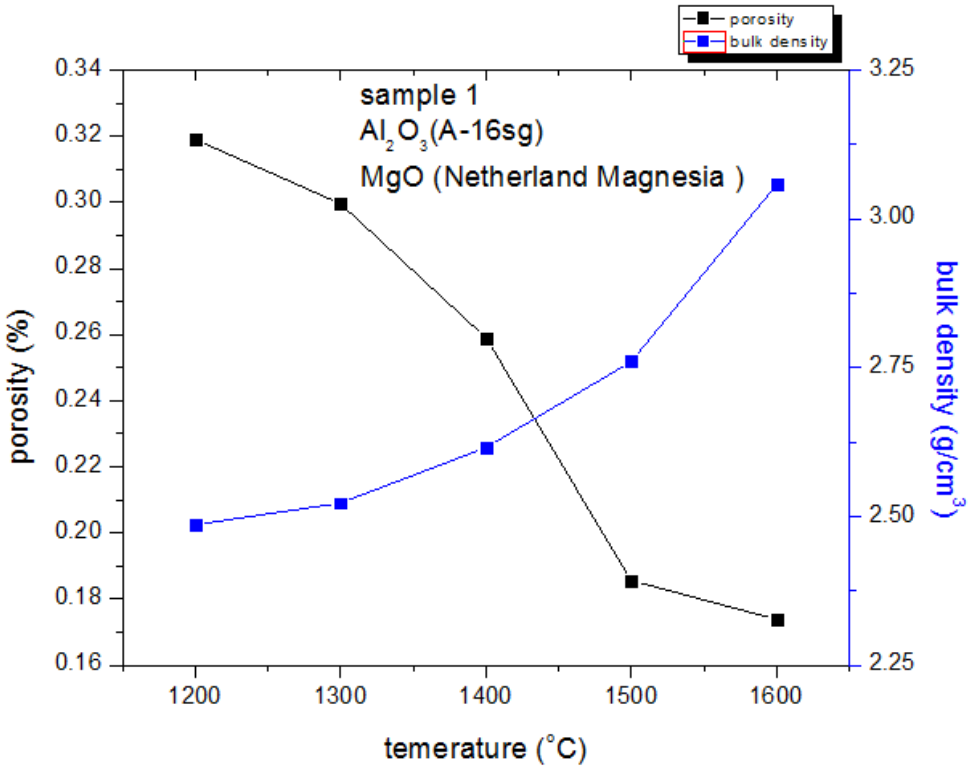
If we compare Batch 1 (A-16sg & Nedmag200) & Batch 2 (CL-370 & Nedmag200) we will observe that sintering is pre dominate at lower temperature for batch1 (1112.9^{o}C) than that of batch 2. As batch 1 contains A16sg which is a reactive alumina which will have property to have high densification. This phenomenon can be seen if compare batch 3&4 or batch 5&6.

If we compare all three type of magnesia we did not see any agreeable effect. Hence we can consider the effect of magnesia is negligible.

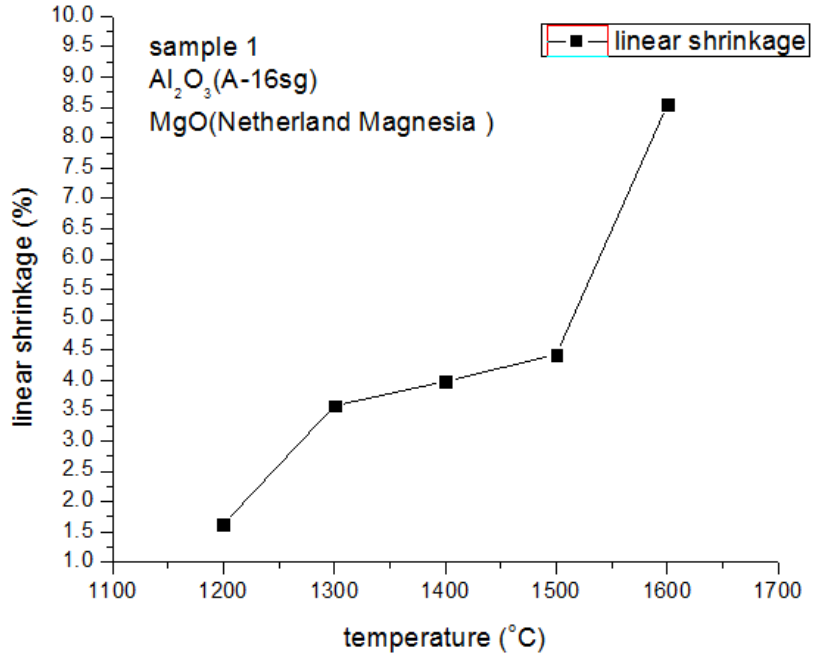
Apparent porosity, bulk density and linear shrinkage

Batch 1 (A-16sg & Nedmag200)

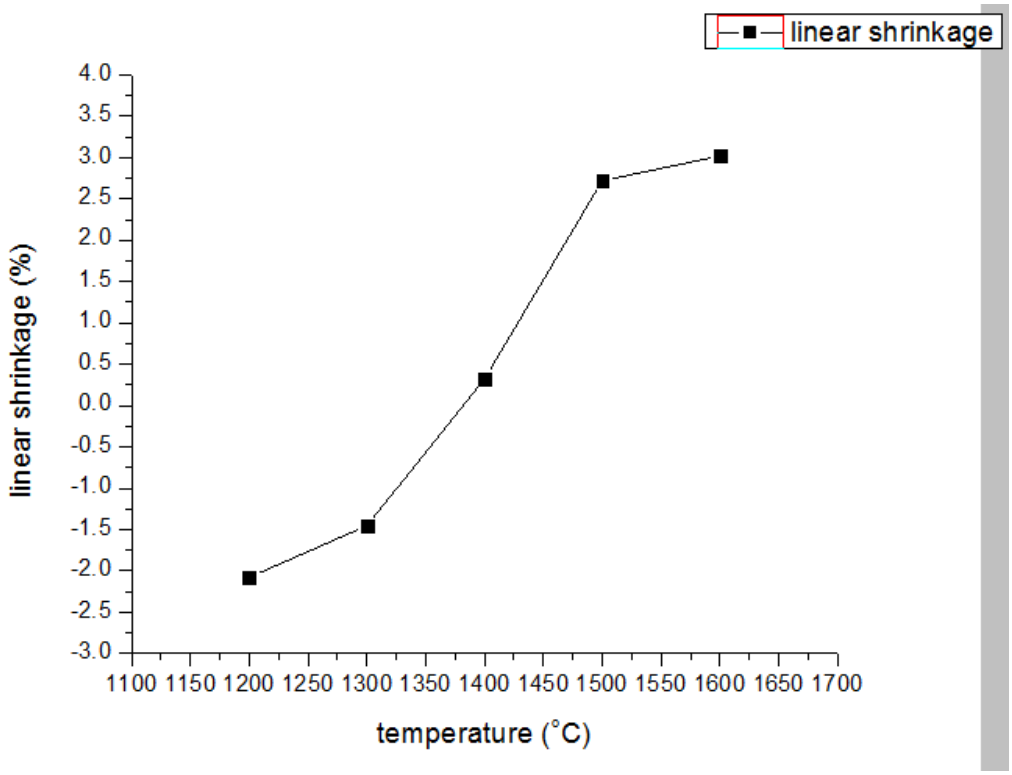
2



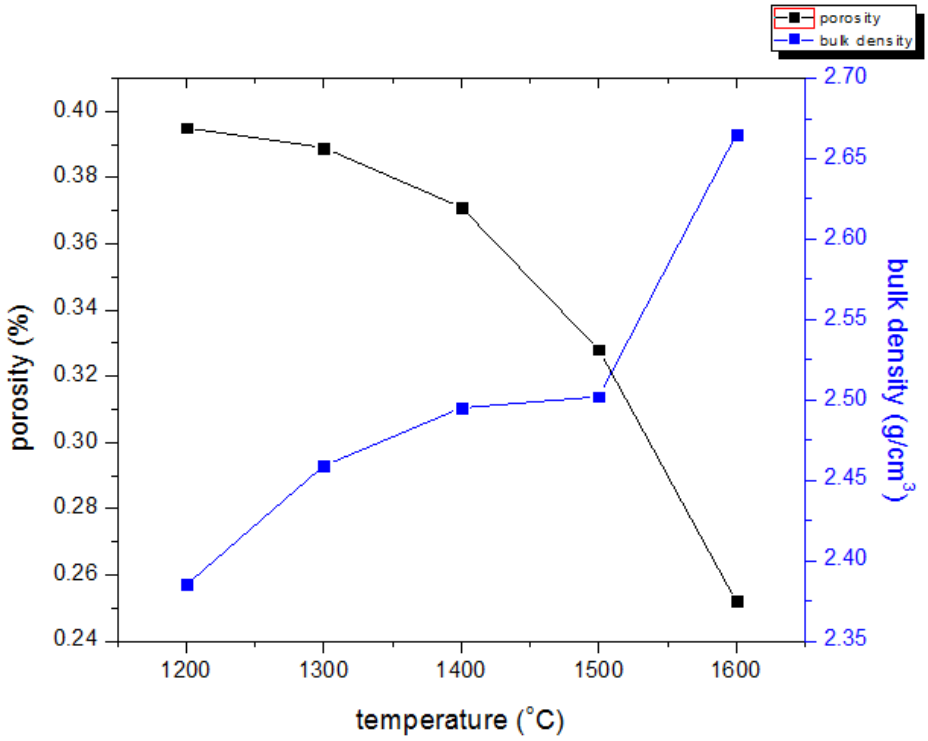
1



Batch 2 (CL-370 & Nedmag200)

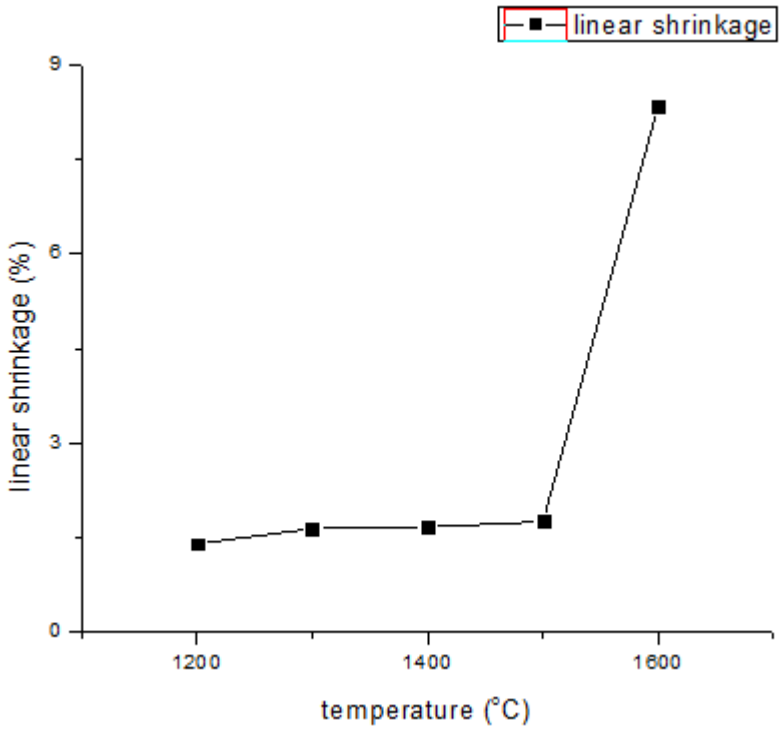


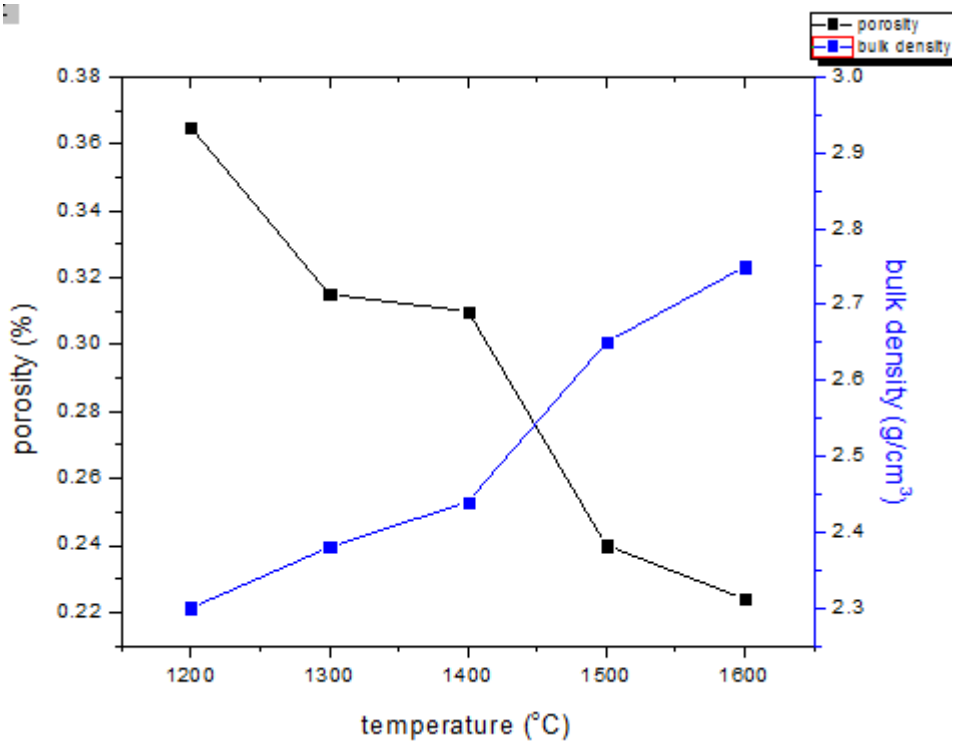
1 2



Batch 3(A-16sg & fused magnesia)

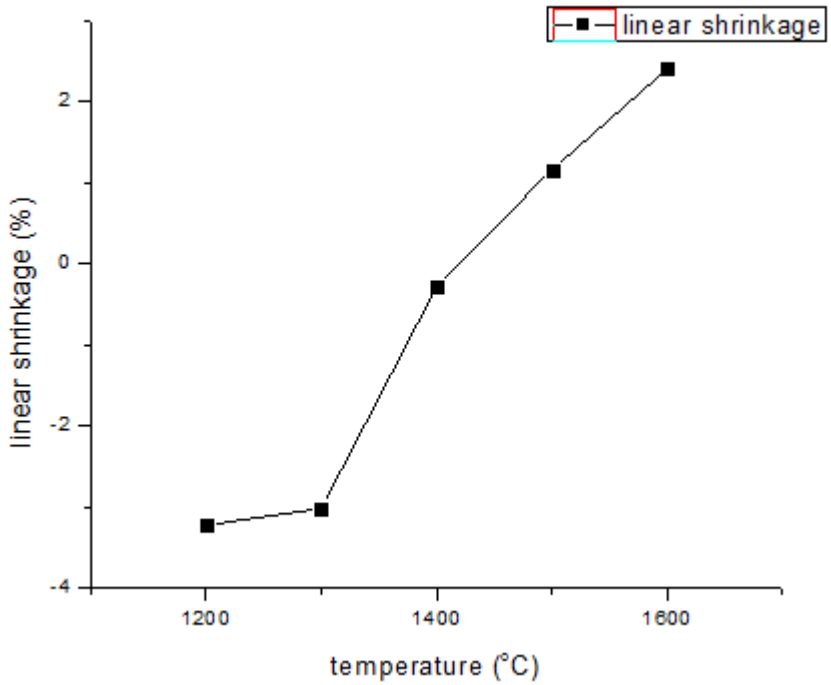
1



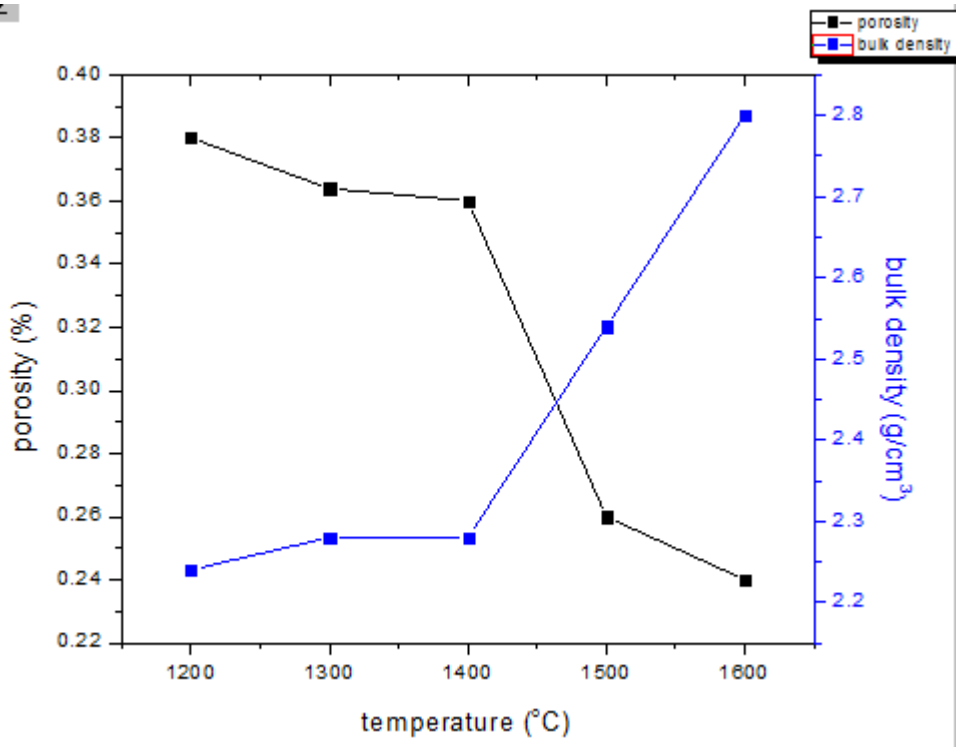


Batch 4(CL-370 & fused magnesia)

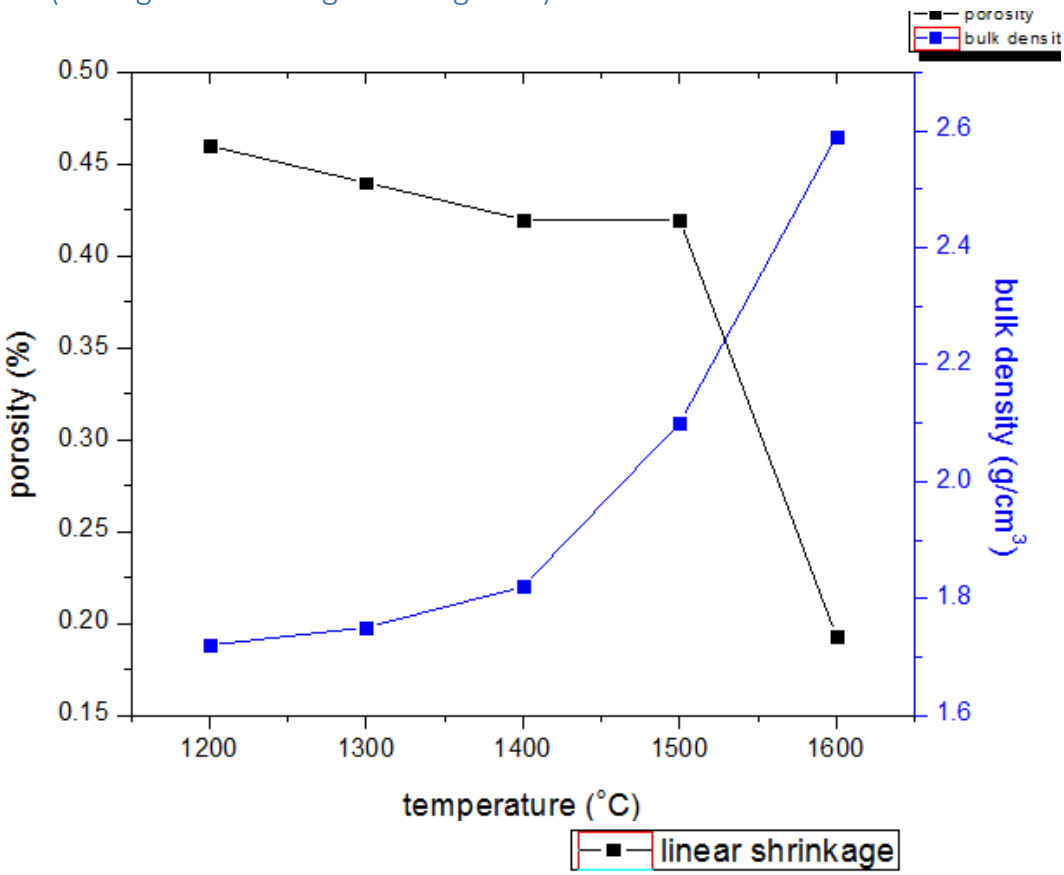
1

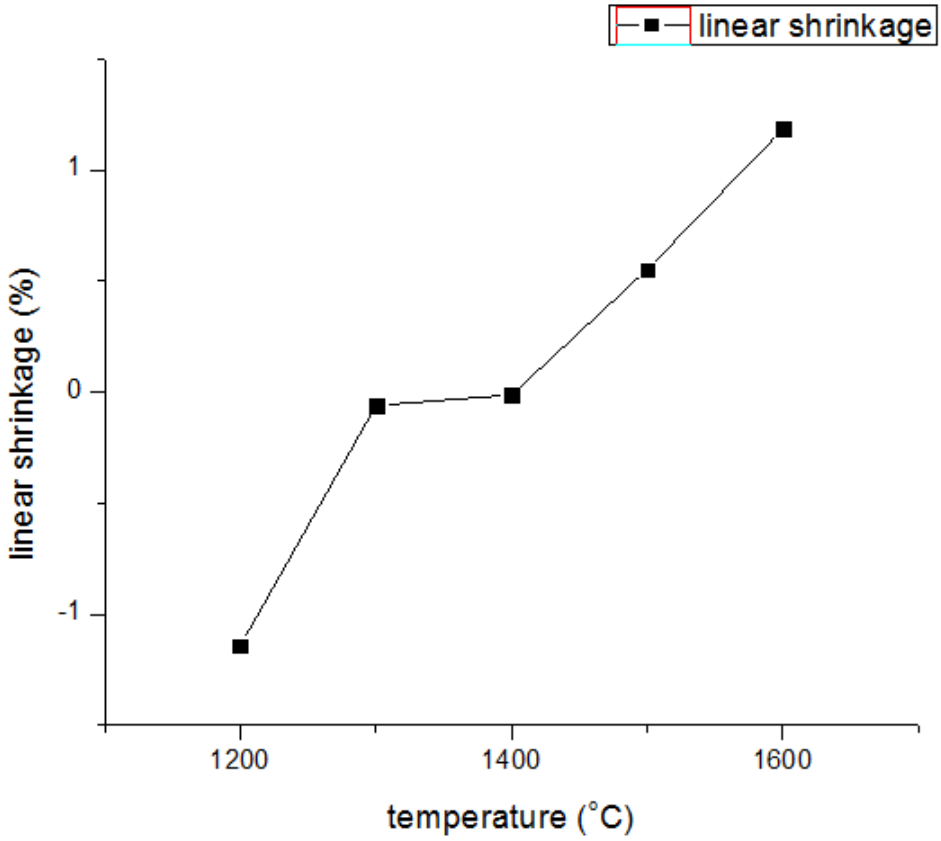


2

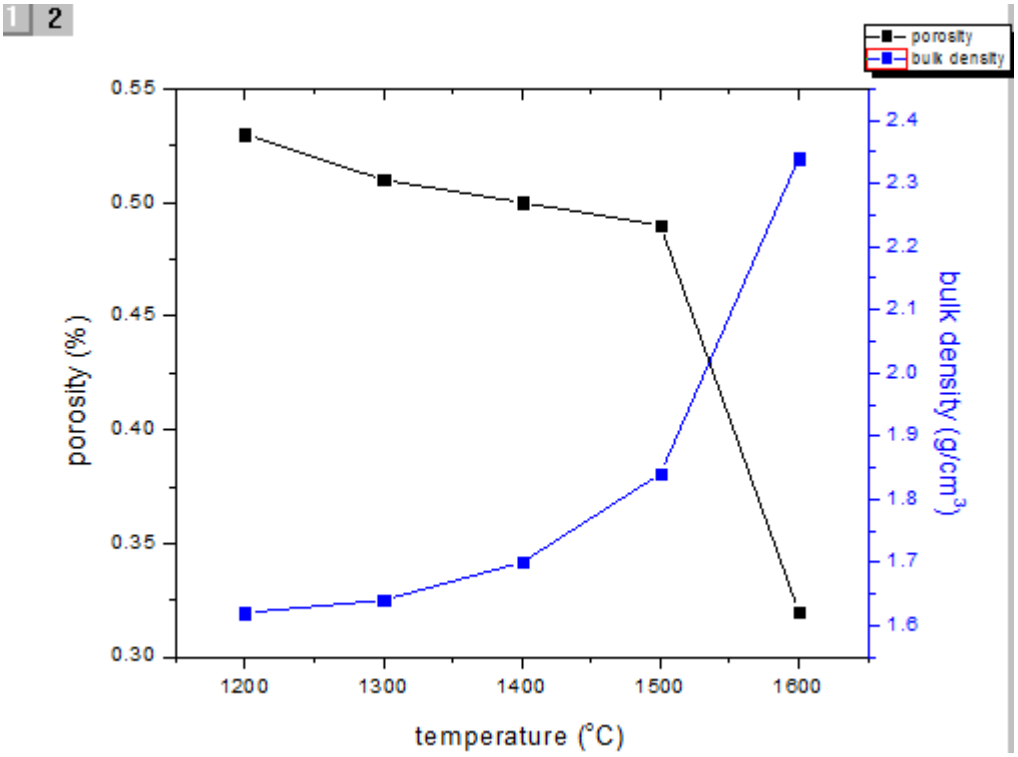


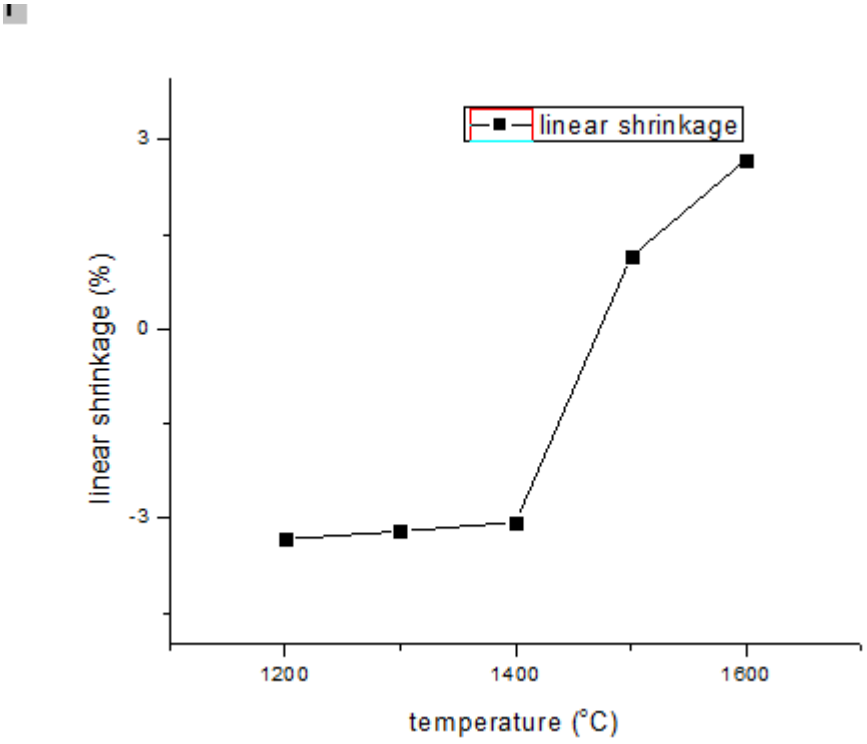
Batch 5 (A16 sg & chemical grade magnesia)





Batch 6 (CL-370 & chemical grade magnesia)





In experiment of apparent porosity and bulk density of all sample, it is observed that with increase in temperature there is increase in the density. The porosity of the sample also decreases with increase in the temperature of the firing. Maximum densification is seen in the sample when fired at temperature above 1500^{o}C. This is because there is complete spinelization of batch at 1500^{o}C hence there is no further expansion. Hence there is high densification is observed due to sintering. Similar way there is maximum linear shrinkage of sample at temperature greater than 1500^{o}C.

Maximum density is observed in batch 1 (Nedmag and A-16sg) where shrinkage starts occurring at the minimum temperature of 1112^{o}C. The density is 3.05g/cm^{3}. And minimum density is observed in batch 6 (CL-370 & chemical grade magnesia) due to formation of porous structure in batch 6 with a sintered density of 2.24 g/cm^{3}.

Scanning electron microscopy images

Batch 1 (A-16sg & Nedmag200)

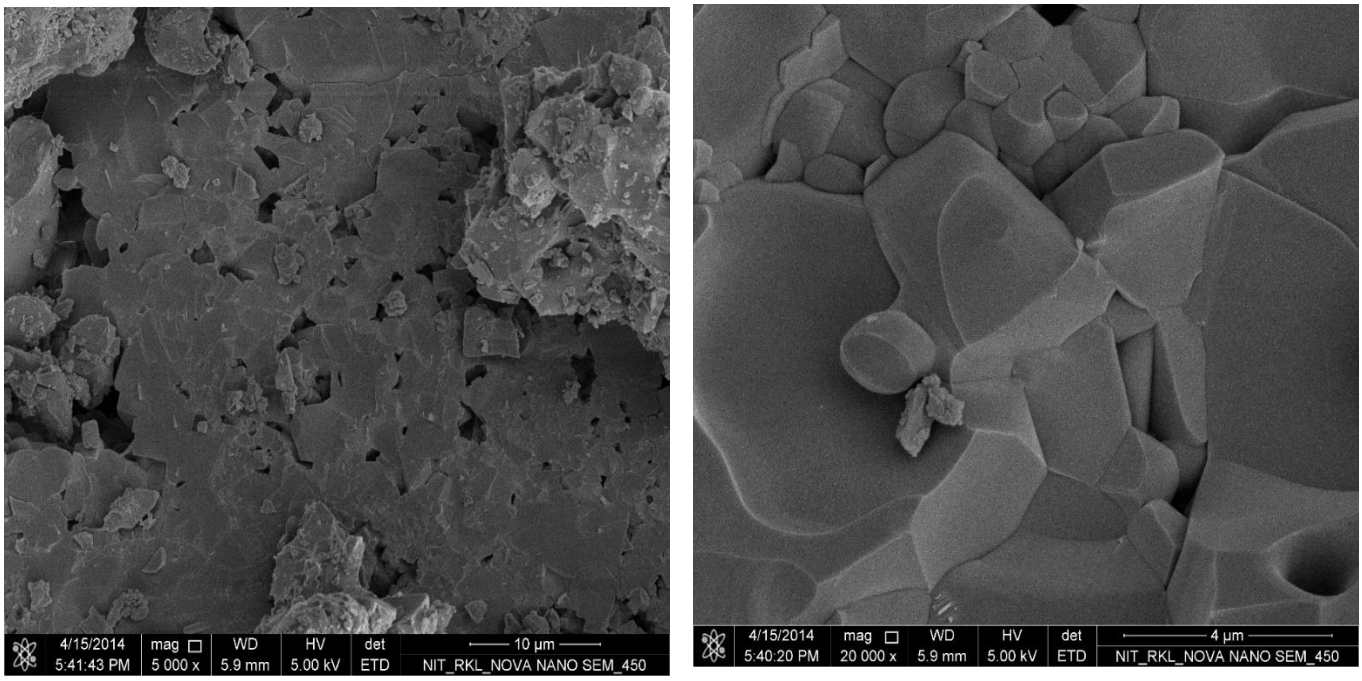


Figure showing SEM image of batch 1 (A-16Sg & Nedmag200 at 1600°C) at 5000x & 20000x respectively

Batch 2 (CL-370 & Nedmag200)

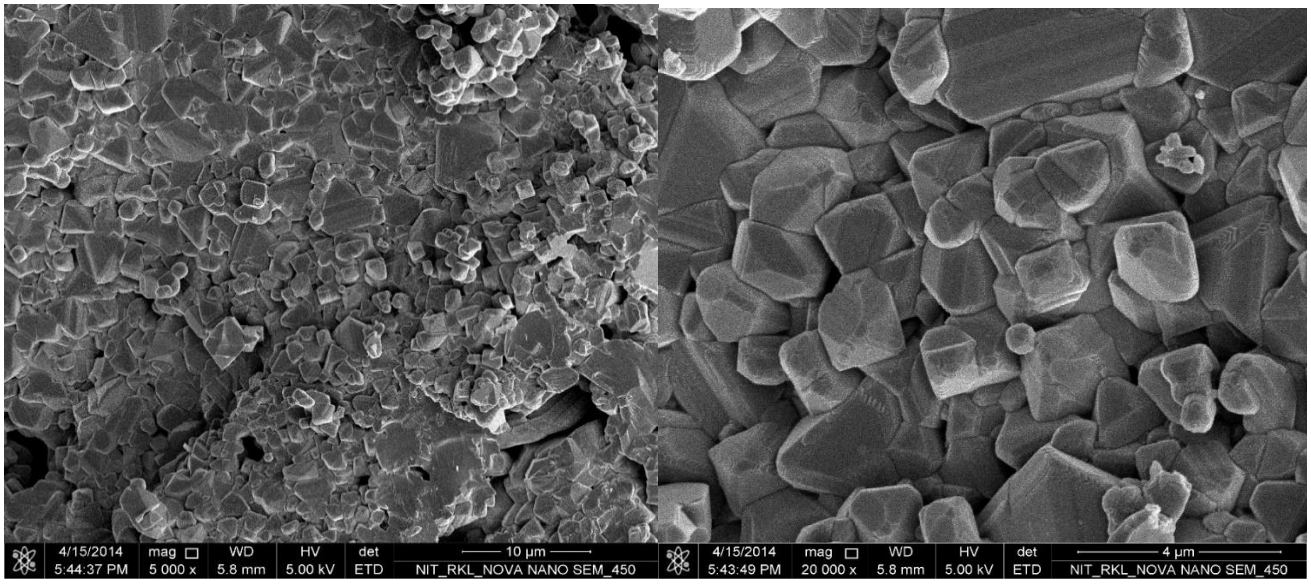


Figure showing SEM image of batch 2 (CL-370 & Nedmag200 at 1600°C) at 5000x & 20000x respectively.

Batch 3 (A-16SG & fused magnesia)

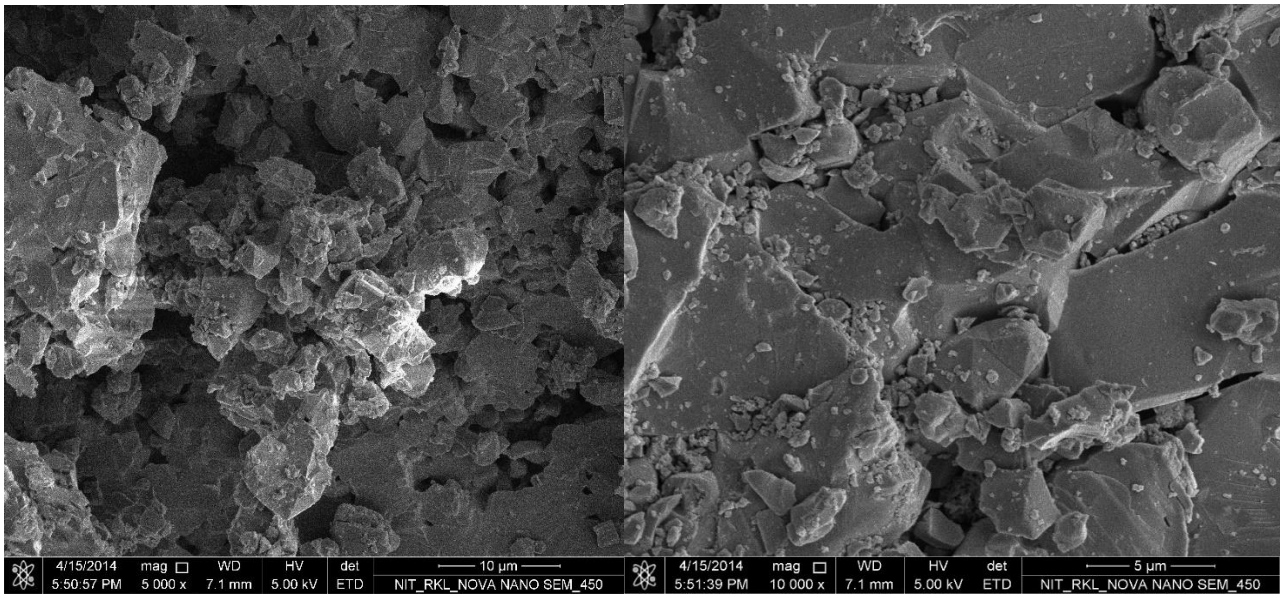


Figure showing SEM image of batch 3 (A-16Sg & fused magnesia at 1600°C) at 5000x & 20000x respectively.

Batch 4 (CL-370 & fused magnesia)

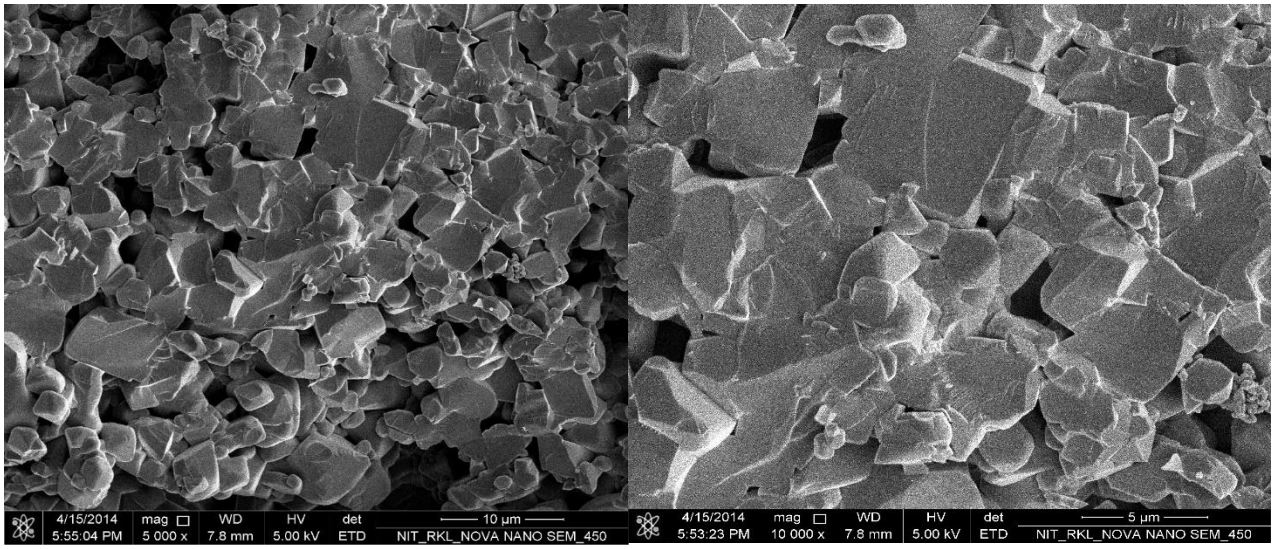


Figure showing SEM image of batch 4 (CL-370 & fused magnesia at 1600°C) at 5000x & 20000x respectively.

Batch 5 (A16-SG & chemical grade magnesia)

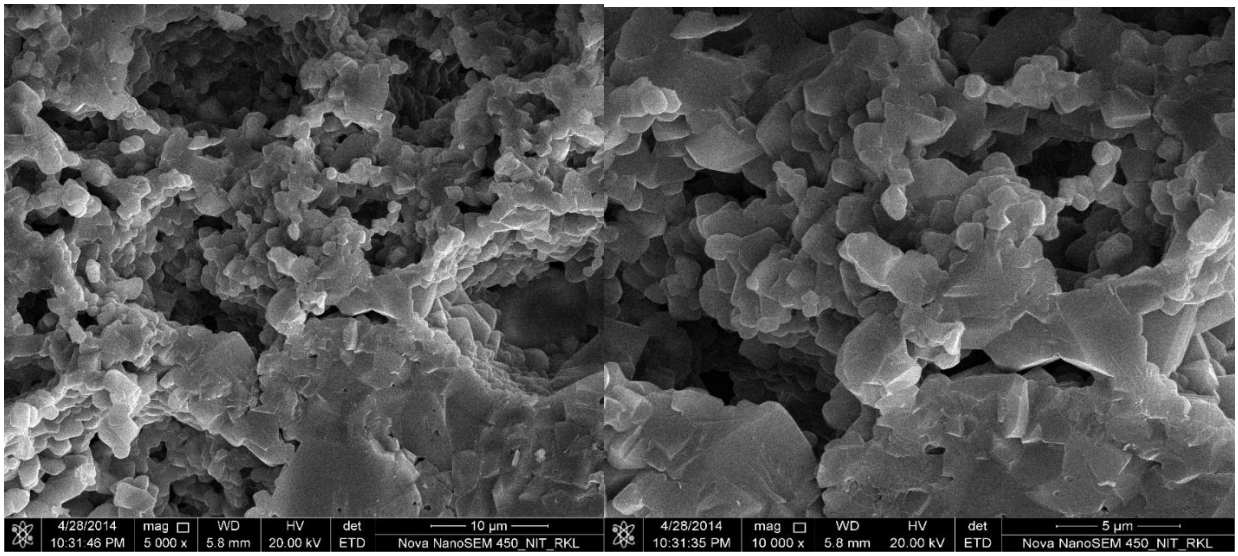


Figure showing SEM image of batch 5 (A16-SG & chemical grade magnesia at 1600°C) at 5000x & 20000x respectively.

Batch 6 (CL-370 & chemical grade magnesia

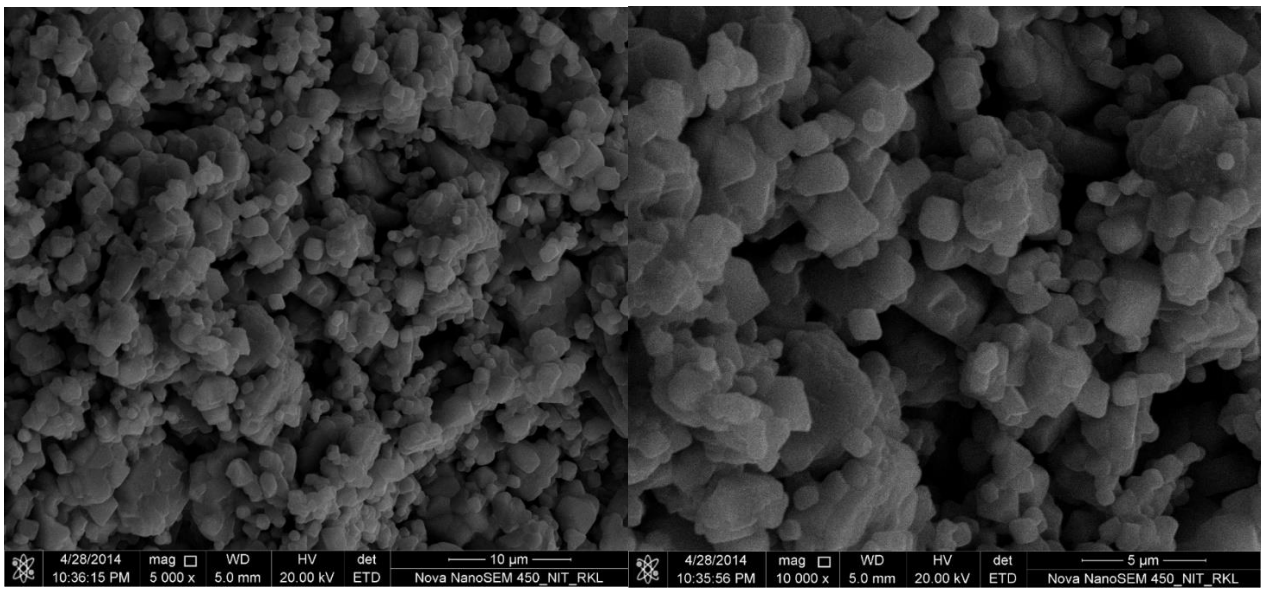


Figure showing SEM image of batch 6 (CL-370 & chemical grade magnesia at 1600°C) at 5000x & 20000x respectively.

Field emission-scanning electron microscopy showed us the microstructure of all the batch. The SEM is done on sample which are fired at 1600^{o}C. The microstructure of each was reviled.

- The high density of batch1 (A-16sg & Nedmag 200) can be seen from the dense microstructure. The grains are larger in size and less porosity then other hence giving high density.
- Batch 6 & batch 5 both (both have chemical grade magnesia) contains fine grain structure which gives high spinel formation but due to small spinel grains creates porous structure.

Conclusion

- The testing of all batches showed the formation of complete spinelization above 1500℃.
    - But composition containing chemical grade magnesia showed complete spinelization above 1500℃. Due to its high surface are.
- The batch in which sintering starts at lower temperature showed high bulk density and low apparent porosity.
- Hence form the experiment we can observe that there is high density and good spinel formation in batch Negmag 200 and A16-sg.
- Composition containing chemical grade magnesia showed spinel formation at lower temperature but failed to achieve good bulk density.
- Chemical grade magnesia containing batches (5&6) showed smaller grain size compared to all other batches.

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